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NICKEL

Rôle of Cathode Nickel in Electron-Tube Performance

J. T. ACKER: 'Cathode Nickel: Its Rôle in Electron-Tube Performance.'

Western Electric Engineer, 1959, vol. 3, Apr., pp. 32–5.

Although electron tubes have been manufactured for nearly half a century, the mechanism by which the oxide-coated cathode emits electrons over a life-span of several years is still not thoroughly understood. It is generally agreed that a small amount of free alkaline-earth metal (barium or strontium) must be formed, by reduction of the oxide coating on the cathode, to produce a semi-conductor which is responsible for electron emission from the cathode, but there are many factors which influence the barium-metal/barium-oxide ratio. Among these factors is the material (usually nickel or a nickel alloy) employed for the cathode core, the effect of which on thermionic emission, particularly in relation to electron tubes manufactured by the Western Electric Company for the Bell System, forms the subject of the present article. The compositions of the various grades of cathode nickel referred to by the author are reproduced in the table below.

Fundamentally the paper comprises a review of past and present work carried out to attain precise control over the cathode material. Until the early 1930's manufacturers of electron tubes generally made use of 'Grade A' nickel, but work to minimise the impurities present resulted in the production, by The International Nickel Company Inc., of '220 Grade' electronic nickel, which has been extensively used as a cathode material in the last twenty years. Other nickel alloys have been used for specific purposes and the author discusses, in this respect, the '225 Grade' (differing in composition from '220 Grade' only in its silicon content: 0·15–0·25 per cent.) which permits rapid activation of the cathode with minimum shrinkage loss due to lack of thermionic emission. The use of silicon as a reducing agent has, however, disadvantages (e.g., the formation of barium orthosilicate, at the interface of the core coating, which impedes the flow of electrons) which have resulted in a trend towards use of very pure nickel and have intensified work to determine additives which would produce quicker activation and greater strength without formation of deleterious reaction compounds. In this connection, the advantages and limitations of a high-purity grade of nickel, designated 'P-50', are discussed and an outline is given of the reasons for, and the problems involved in, the substitution of this grade of nickel for the '220 Grade' used in electron tubes manufactured by the Western Electric Company. To compare the performance of cathodes manufactured from each of these grades of nickel, life tests were made, under cyclic conditions, on 404A and 417A electron tubes. The data presented indicate the superior performance of the 'P-50' grade.

In field trials with 'P-50' cathodes it was observed that the transconductance tended to increase during the first 100 hours of life. This defect was overcome by introducing a longer stability ageing treatment during manufacture, a treatment, however, which increased the cost of the tube. Confronted with this problem, and with the need for better control over the composition of the cathode nickel, the Bell Telephone Laboratories initiated work to produce, from carbonyl nickel, an improved cathode material for use in long-life tubes. Notes on the progress of this work are given. On a production scale the carbonyl-nickel powder will be subjected first to a low-temperature sintering heat-treatment, in hydrogen, to remove the carbon and oxygen present. The sintered nickel will then be melted in a hydrogen

Composition of Various Grades of Cathode Nickel

Type	Cu %	Mn %	Fe %	C %	Mg %	Si %	Ti %	S %	Ni + Co %
'Grade A' Nickel	0·20 max.	0·35 max.	0·30 max.	0·20 max.	Not specified	0·20 max.	Not specified	0·008 max.	99·00 min.
'220 Grade' Nickel	0·20 max.	0·20 max.	0·20 max.	0·08 max.	0·01-0·10	0·01-0·05	Not specified	0·008 max.	99·10 min.
'P-50' Nickel	0·04 max.	0·02 max.	0·05 max.	0·05 max.	0·01 max.	0·02 max.*	0·01 max.	0·005 max.	99·50 min.

* '499' nickel differs from 'P-50' nickel only in silicon content, which is specified at 0·01 max. per cent.

atmosphere, which will be removed by evacuation and replaced by helium. Magnesium, in the form of a nickel-magnesium alloy, and pure tungsten rods will be added to the molten nickel under the helium atmosphere, and, after thorough diffusion, the melt will be cast into a refractory-coated steel mould. Special metallurgical precautions will be taken to ensure that no other impurities are introduced during melting and casting. The final composition of the initial nickel melts of this type will contain 2 per cent. tungsten and 0·025 per cent. magnesium. Cathodes will be fabricated from the alloy in the usual manner. Magnesium was selected as an additive because of its excellent reducing effect on the alkaline-earth oxides and because it apparently does not form an interface impedance compound on the cathode-nickel surface. The addition of tungsten strengthens the nickel and is believed to have a mild reducing effect on alkaline-earth oxides. The high purity of the nickel minimises the risk of formation of interface compounds by impurity reactions.

Electrical-Resistivity Recovery in Cold-Worked and Electron-Irradiated Nickel

A. SOSIN and J. A. BRINKMAN: 'Electrical-Resistivity Recovery in Cold-Worked and Electron-Irradiated Nickel.'

Acta Metallurgica, 1959, vol. 7, July, pp. 478–94.

Following work by CLAREBROUGH *et al.* on the release of stored energy in cold-worked pure and arsenic-doped copper and in relatively pure nickel (references to which are given and the results of which are summarised in the present paper), the authors made a detailed study (over the range room temperature to 300°C.) of the kinetics of recovery in nickel wire after cold-working, and, as a further aid to interpretation of the recovery process, after irradiation with 1·25 MeV electrons.

CLAREBROUGH *et al.* found that two distinct stages are involved in the recovery of nickel: a lower recovery stage, possibly associated with the migration and subsequent annihilation of vacancies, and an upper stage attributable to recrystallisation. In the investigation now reported recovery stages were found to occur, after cold-work, by a diffusion process in the region of 100°C. (Stage III) and 270°C. (Stage IV); dislocations are believed to be the defect sinks. Following irradiation, Stage III was found to obey a second-order chemical rate equation; Stage IV was essentially absent. An activation energy for defect migration of about 1·5 eV was determined in Stage III both after irradiation and cold-work; the energy associated with defect migration in Stage IV was not as well determined.

These measurements, together with other relevant data available on nickel, indicate that the defect migrating in Stage III was an interstitial atom and in Stage IV a vacancy. The close similarity between the present results and data previously obtained for copper suggests that a similar assignment may be applied to the latter metal.

Influence of Chromium on Strain Hardening and Softening of Nickel

M. P. ARBUZOV and M. P. KRULIKOVSKAYA: 'Effect of Chromium on (Strain) Hardening and Softening of Nickel.'

Fiz. Metal. i Metalloved, 1958, vol. 6, pp. 1070-6.

The work described was carried out to determine the influence (as reflected in changes in lattice distortion, size of mosaic blocks and hardness) of alloying additions of 3·87, 7·65, 14·43 and 18·9 per cent. of chromium on the strain-hardening and softening characteristics of nickel. The alloys were cold-worked to 80 per cent. compression and softening was carried out at temperatures in the range 400°–850°C. For a summary of the salient findings see *Chemical Abstracts*, 1959, vol. 53, p. 12992.

Dispersion-Hardening of Nickel with Aluminium Oxide

See abstract on p. 332.

Direct Electron-Microscopical Examination of Nickel Foils

See abstract on p. 343.

Joining of Nickel to Other Materials

See abstract on p. 344.

ELECTRODEPOSITION AND OTHER COATING METHODS

Decorative High-Chloride Nickel Plating

M. TIPPMAN: 'Decorative High-Chloride Nickel Plating.'

Plating, 1959, vol. 46, Sept., pp. 1054–6.

It is the author's conviction that nickel coatings produced from the high-chloride modification of the Watts solution, using the operating conditions described in the paper, are equivalent in every respect to those deposited from any other plating solution commercially available. The high-chloride solution, it is claimed, performs outstandingly well from the point of view of quality, speed of deposition and economy. It obviates the need for blowers, air-agitation piping or special high-temperature linings, results in savings in power and drag-out, and, because of excellent anode corrosion, minimises the need for salt replenishment. In this paper the author summarises experience gained in the operation of this type of plating solution.

For decorative applications, and to obtain optimum corrosion-resistance, the author recommends use of the following solution composition and plating conditions:

	oz./gal.	*g./L.*
Nickel metal ..	9–10	56·1–62·4
Nickel chloride ..	12–25	74·8–156
Boric acid	6	37·4
pH	4·0–4·8	
Cathode current density	45 *amp./sq. ft. and higher* 4·86 *amp./dm.²* *and higher*	
Temperature	145–150°F. 63–65°C.	

Addition agents should be used in accordance with the supplier's recommendation. The nickel-sulphate concentration is adjusted to furnish the balance of the nickel metal.

Factors influencing the operation of the solution and the properties of the deposit are discussed in sections relating to chemicals, anodes, treatment of the basis metal prior to plating, solution operation, nickel coverage and prevention of roughness, cloudy deposits, corrosion-resistance, conversion of a Watts system to high-chloride plating, treatment of the solution, and brighteners.

Influence of Fatty Acid Films on the Adhesion of Nickel Deposits

H. B. LINFORD and J. J. GRUNWALD: 'A.E.S. Research Project No. 12. Cleaning and Preparation of Metals Prior to Electroplating. XII. Effect of Fatty-Acid Film.'

Plating, 1959, vol. 46, Sept., pp. 1039–45.

The work described was undertaken with the aim of obtaining quantitative data on the effects of greasy impurities on the adhesion of nickel deposits to copper. The paper is introduced by a survey of the relevant literature.

Stearic-acid films were deposited, using three different techniques, on the surface of 'atomiser-clean' copper specimens. The specimens were then nickel plated, and the adhesion of the coatings was evaluated as a function of the amount of stearic acid present on the basis metal. From the data obtained it is concluded that organic contaminants which cannot be detected by a correctly performed 'water-break' test have no effect on adhesion. The presence on the copper of even small traces (equivalent to $<0{\cdot}05$ of a monolayer) of such contaminants will result in pitting due to hydrogen-bubble attachment. Organic contamination of the cathode was found to cause preferential discharge of hydrogen during the first fractions of a second of nickel plating.

Influence of Wetting Agents on the Hardness of Nickel Deposits

H. SPÄHN: 'The Influence of Wetting Agents on the Hardness of Electrodeposited Nickel Coatings.'

Metalloberfläche, 1959, vol. 13, Aug., pp. 259–64.

Previous work to determine the influence of solution composition and plating conditions on the properties of nickel electrodeposits having been carried out mainly in the absence of wetting agents, the present investigation was initiated to establish the influence of such additions on the hardness (the property most easily determined) of nickel coatings.

The following wetting agents were selected for study: lauryl sulphate, coconut-oil-alcohol sulphate+dodecylbenzyl sulphonate, lauryl diglycolether sulphate, coconut-oil-alcohol polyglycolether sulphate+coconut-oil-alcohol polyglycolether, coconut-oil-alcohol polyglycolether phosphate, and coconut-oil-alcohol polyglycolether. The electrolyte used in the investigation was of the following composition: $NiSO_4.7H_2O$ 220, $NiCl_2.6H_2O$ 30, H_3BO_3 30, g./L. In general, plating was carried out at 40°C., using a current density of 4 amp./dm.^{2} and a pH of from 4·6 to 4·7. The coatings were electrolytically polished before micro-hardness testing.

Addition of small amounts of anion-active fatty alcohol sulphate (lauryl sulphate) caused a decrease in hardness from 340 to 190 kg./mm.^{2}. Additions of from 300 mg./L. of lauryl sulphate inhibited pore formation. Deposition of nickel progressed normally at all the concentrations studied.

Purely aliphatic anion-active wetting agents with ether bridges and containing a minimum amount of oxygen heteroatoms (lauryl diglycolether sulphate) had a similar effect on hardness and ductility to that produced by the fatty alcohol sulphates. When the compounds contained numerous oxygen bridges with affinity to water, however, their behaviour during nickel plating resembled that of the non-ionic wetting agents.

Of the latter class of materials, an alkylpolyglycolether (coconut-oil-alcohol polyglycolether) was studied. Even small additions of this substance resulted in a considerable increase in hardness, and at concentrations above 0·2 ml./L. the deposits were brittle and technically unusable. In contrast to the fatty alcohol sulphates, such non-ionic wetting agents were obviously incorporated in the deposit even at small concentrations.

The variations in hardness discussed are considered to be of relevance to practical nickel plating in several respects: the decrease in hardness is, for example, of importance in relation to the corresponding increase in ductility, and the increase in hardness resulting from addition of non-ionic wetting agents is of significance in relation to hard-nickel plating.

Sensitivity of the Ferroxyl Porosity Test

F. OGBURN, D. W. ERNST and W. H. ROBERTS: 'A.E.S. Research Project No. 13. The Nature, Cause and Effect of the Porosity in Electrodeposits. V. An Evaluation of the Sensitivity of the Ferroxyl Test.'

Plating, 1959, vol. 46, Sept., pp. 1052–3.

In its original form the ferroxyl test, one of the main methods of determining porosity in nickel coatings, involved the application to the coating of a solution of sodium chloride and potassium ferricyanide, a blue spot being formed wherever the basis metal was exposed. Several investigators have, however, shown that this reagent attacks the nickel coating and may, as a result, itself produce pores. Strausser modified the test to obviate such attack on the coating by eliminating any contact between the ferricyanide and the nickel. In the present investigation the reliability of the Strausser procedure was assessed on the basis of its efficacy in determining porosity in gold coatings on steel. After testing, the steel was dissolved and the isolated coating was examined microscopically and photographically for pores. This examination, which enabled an

estimate to be made of the size of each pore corresponding to a ferroxyl spot, showed that the Strausser version of the test detected less than 50 per cent. of the pores present with diameters of from 0·05–0·5 mil., and that the larger pores were detected more consistently than the smaller ones.

(For abstracts of previous reports of the project see *Nickel Bulletin*, 1959, vol. 32, No. 9-10, pp. 282–3.)

Corrosion-Resistance of Nickel/Chromium Deposits in Automotive Applications

'Chromium Plating. Parts I and II.'

Autocar, 1959, vol. 111, Sept. 18, pp. 210–11; Oct. 9, pp. 328–9.

The article reviews the factors underlining the apparent deterioration in nickel/chromium coatings on car trim since pre-war days and discusses the efforts which are being made to increase corrosion-resistance.

In the introductory section the assertation is made that 'though chromium plating in car trim may not be as good as it could be, those who are best qualified to know declare that it is better in all respects to-day than it has ever been. But cars nowadays have up to ten times as great a surface area of such brightware as in pre-war days, there are many more cars—and naturally there are more complaints. To some extent, current criticisms of faulty plating are based on the evidence to be seen on cars now several years old, the plating of which was done in the lean years when nickel was rationed because of an acute world shortage, and it was impossible to apply a sufficient thickness of this basic protection. In the last year or two supplies of nickel have eased, and platers can apply an adequate thickness, the benefits of which will be seen in the future.'

Other sources of complaint can be ascribed to the more arduous conditions of service (e.g., as a result of the great increase in atmospheric pollution), to the greater complexity of shapes which renders sound plating more difficult, and, in some cases, to the lower quality of the metal forming the basis material. These points are enlarged upon, and reference is also made to the importance of the nickel layer in conferring corrosion-resistance on the composite coating. In the latter connection attention is drawn to a projected revision of the relevant British Standard which, for certain applications, stipulates increased minimum thicknesses for the nickel layer (the requirements of the existing and projected standards are summarised in tables).

Part II reviews recent developments in nickel and chromium plating which facilitate deposition of the two metals or offer promise of considerably improved corrosion-resistance. The advantages of automatic plating are briefly considered, and reference is made to two innovations in nickel and chromium plating: duplex nickel coatings, and duplex and crack-free chromium coatings. (For further information on these two developments see following abstract.) Reviewing the position with respect to corrosion-resistance, however, the author emphasises that the standard of quality of plated coatings depends above all on the observance, both by platers and car manufacturers, of the requirements laid down by the relevant British Standard.

Corrosion-Resistance of Duplex and Crack-Free Chromium Deposits

D. E. WEIMER: 'Crack-Free and Duplex Chromium: Answers to Better Corrosion-Resistance.'

Electroplating and Metal Finishing, 1959, vol. 12, Sept., pp. 340–3, 348.

Work carried out with the aim of improving the corrosion-resistance of nickel/chromium electrodeposits has shown that duplex nickel coatings (comprising an underlayer of semi-bright nickel and a second layer of bright nickel) exhibit a much higher corrosion-resistance compared with that of conventional bright deposits (see, for example, the article by CALDWELL abstracted in *Nickel Bulletin*, 1959, vol. 32, No. 8, pp. 251–2). Research on similar lines has now been conducted on chromium deposits. This work is described in the present paper.

Crack-free chromium deposits have been obtainable, under specific plating conditions, for some years (those discussed by the author are deposited from a proprietary solution, the 'CF-520'). Such deposits are crack-free, however, only up to a thickness of 0·1 mil, and in many applications a minimum of 0·02 mil cannot be exceeded without cracking at the highest-current-density areas. To increase the thickness further, the crack-free underlayer is coated (using a 'CR-110' solution) with a second deposit, having a controlled crack pattern, which raises the minimum thickness of chromium in the recesses of the basis metal to at least 0·03 mil and the average thickness of the chromium to 0·1 mil. The first chromium deposit provides optimum coverage in high-current-density areas. The second, as a result of its many cracks, disperses the cell action over a large area and ensures that the corrosion current density is maintained below the critical potential required for anodic perforation of the underlying metal. The process is claimed to be adaptable, with minor modification, to most existing plating operations.

Data are tabulated to indicate the relative corrosion-resistance (as assessed by the 'Corrodkote' and copper-accelerated acetic-acid/salt-spray tests) of copper/nickel/duplex-chromium coatings of various thickness.

Electrodeposition of Chromium Alloys: Literature Review

C. LEVY: 'Electrodeposition of Chromium Alloys: A Literature Review.'

Metal Finishing, 1959, vol. 57, Aug., pp. 59–62, 65.

In the belief that the solution to the problem of cracking in chromium electrodeposits lies in the development of simple and economic methods of depositing chromium-alloy coatings with chemical and physical properties equal or superior to those of

chromium deposits, the author carried out an extensive literature survey to determine methods already available for this purpose. His findings are summarised in the present paper.

The review, which is based on a *bibliography* of 67 items, covers literature relating to solutions used to deposit chromium-iron, chromium-nickel, chromium-cobalt, chromium-tungsten and chromium-molybdenum alloys, and miscellaneous binary and ternary chromium alloys.

NON-FERROUS ALLOYS

Influence of Nickel on the Age-Hardenability of Copper-Beryllium Alloys

H. THOMAS and U. WILKE-DÖRFURT: 'The Influence of Alloying Additions on the Age-Hardenability of Copper-Beryllium Alloys.'

Zeitsch. f. Metallkunde, 1959, vol. 50, Aug., pp. 466–72.

A study was made of the influence of the following alloying additions (introduced as a third element in the amount stated) on the age-hardening characteristics of a copper-beryllium alloy containing 2 per cent. beryllium: iron, aluminium, silicon, nickel, chromium (0·3 per cent.); cobalt (0·2 per cent.); manganese (0·4 per cent.).

Specimens of each alloy were homogenised at 700°, 750°, 800° or 840°C., water-quenched, and then aged at 200°, 250°, 300°, 350° or 400°C. The rate and mechanism of the age-hardening process were investigated (as a function of solution-treatment and time and temperature of ageing) by determination of changes in Vickers hardness and electrical resistance, and by metallographic examination.

It is concluded from the data obtained that, at ageing temperatures up to 300°C., cobalt and nickel additions greatly decrease the rate of age-hardening but increase the maximum hardness attained. Iron has a similar effect only when the alloy is homogenised at temperatures of 800°C. and above, and manganese only at ageing temperatures below 250°C. Aluminium, silicon and chromium have no significant effect on the rate of age-hardening.

The influence of the additions on the precipitation mechanism is explained in terms of atom size and their solubility in the matrix: those with small atomic radii facilitate the formation of the intermediate tetragonal phase γ'; those with large atomic radii strengthen the tendency to direct precipitation of the γ phase.

Influence of Alloying Additions on the Recrystallisation of a Nickel-Cobalt Alloy

S. FLOREEN: 'The Effect of Alloying Elements on the Recrystallisation of a Nickel-10% Cobalt Alloy.'

Jnl. Inst. Metals, 1959, vol. 87, July, p. 384.

Previous investigations have shown that very small additions of solute atoms considerably increased the softening temperature of a pure metal, a result which has been attributed to the concentration of solutes at the grain boundaries, where they impede recrystallisation. The study reported in this short note was carried out to determine the effect of larger additions of solute elements on the recrystallisation temperature of a nickel-cobalt alloy containing 10 per cent. cobalt.

A series of ternary alloys was produced by adding to the basis alloy the following percentages of aluminium, copper, or chromium: aluminium 3·42, 5·42, 7·63, per cent.; copper 1·85, 5·47, 11·17, per cent.; chromium 2·39, 6·67, 14·22, per cent. Ingots melted and cast in an argon atmosphere were machined into strips, which were then cold-rolled 10 per cent., annealed at 1600°F. (870°C.), cold-rolled 10 per cent., annealed at 1400°F. (760°C.), and finally cold-rolled 20 per cent. These specimens were annealed at various temperatures for 6, 24 and 96 hours. The recrystallisation temperatures determined (primarily by means of hardness measurements) are tabulated.

The recrystallisation temperature of the alloy increased with increasing additions of chromium, but decreased, from an initially high level, as the copper content was increased. The recrystallisation temperature of the aluminium-containing alloys was at a minimum with a content of 5·42 atom per cent. aluminium. It is emphasised that the recrystallisation temperature of all the alloys studied was well above that of the nickel-cobalt basis composition.

Heats of Formation of Copper-Nickel Alloys

J. S. LI. LEACH and M. B. BEVER: 'On the Heats of Formation of Copper-Nickel Alloys.'

Trans. Metallurgical Soc. Amer. Inst. Mechanical Engineers, 1959, vol. 215, Aug., pp. 728–9.

The heat effects on dissolution at 350°C. in tin were measured calorimetrically for copper, nickel, and copper-nickel alloys containing 12, 20, 32 and 40 at. per cent. nickel. Using these data the heats of formation of the copper-nickel alloys were calculated. The values obtained, together with electrochemical values, are reproduced in the table below.

A positive heat of formation of the magnitude obtained for these alloys is considered to support evidence in the literature indicating clustering or a miscibility gap at low temperatures.

Heats of Formation of Copper-Nickel Alloys

Alloy Composition	Calorimetric Value at 0°C., Cal. per G Atom	Electrochemical Value at 700°C., Cal. per G Atom
88 Cu-12 Ni	$+640 \pm 155$	$+360$
80 Cu-20 Ni	$+740 \pm 275$	$+540$
68 Cu-32 Ni	$+895 \pm 485$	$+740$
60 Cu-40 Ni	$+940 \pm 395$	$+810$

Direct Electron-Microscopical Examination of Thin Alloy Foils

See abstract on p. 343.

Decorative Use of Sintered Nickel Silver Components

'Nickel Silver Sinterings.'

Metal Industry, 1959, vol. 95, Sept. 18, pp. 121–2.

Due to the limited degree of lateral flow which can occur in the metal powder during compacting, sintered components tend to be angular in character, and since the trend of industrial design has in the past been towards smoothly rounded contours, almost all the applications of powder-metallurgically-produced components have been purely utilitarian. In general, therefore, such parts have not required any surface-finishing treatment. There are, however, indications that a new hardness of line is beginning to find favour in industrial design, and that wider use will be made of powder metallurgy in the production of exposed functional components. This trend has concentrated attention on the development of metal-finishing techniques suitable for sintered parts. The techniques available are discussed in this brief article.

Vacuum-metallising and application of organic finishes present no difficulties, but electroplating is less suitable due to risk of corrosion from the electrolyte. Particular interest has been shown, therefore, in those parts which can be given a buffed finish comparable to that of an electrodeposited coating and which have a relatively high impact-resistance. These requirements are met by sintered copper-nickel-zinc components which, it is stated, may be given a high-quality finish merely by barrel burnishing, scratch brushing or buffing.

Fusion Welding of Non-Ferrous Alloys

J. G. YOUNG: 'Progress in the Fusion Welding of Non-Ferrous Alloys.'

Sheet Metal Industries, 1959, vol. 36, Aug./Sept., pp. 557–66, 576.

The majority of the important developments which have transformed the welding of non-ferrous materials from an art to a science, including the introduction of the inert-gas-shielded arc processes, have taken place since the end of the last war. The purpose of this paper is to draw attention to these developments and to review the present state of fusion-welding technology.

The paper is in three sections. In the first the author refers to the manual and mechanised welding processes available, and comments on the scope of, and the equipment used for, each process. The manual processes discussed are: oxyacetylene welding, metal-arc welding, argon-arc welding, and inert-gas metal-arc welding (the various names used to describe which are listed). Notes on mechanised welding relate to the argon-arc and inert-gas metal-arc processes.

The second, and main, section of the review is concerned with the metallurgical factors governing selection of the most suitable welding technique and filler wire for particular sheet materials. No attempt is made to give details of welding conditions since these are readily available elsewhere, but reference is made, wherever possible, to the relevant British Standards and to the composition of the filler wires covered. Information is presented on the welding of aluminium and its alloys, copper and its alloys (copper-aluminium, copper-nickel, copper-silicon, copper-tin, copper-zinc and high-copper alloys); lead and lead-rich alloys; magnesium and its alloys; nickel and its alloys; and titanium.

In the last section the author briefly outlines developments in the welding of non-ferrous alloys which might reasonably be anticipated in the coming decade: extension of the scope of the inert-gas metal-arc process; use of finer wires and of welding currents other than d.c. electrode positive; and improvements in the welding characteristics of non-ferrous materials.

Joining of Nickel-containing Alloys to Dissimilar Materials

See abstract on p. 344.

Non-Ferrous Bourdon Tubing: British Standard

See abstract on p. 330.

CONSTRUCTIONAL STEELS

Influence of Sharp Cracks on the Brittle-Fracture Characteristics of Nickel-Molybdenum-Vanadium Forging Steel

E. T. WESSEL: 'The Influence of Pre-Existing Sharp Cracks on Brittle Fracture of a Nickel-Molybdenum-Vanadium Forging Steel.'

Amer. Soc. Metals, 1959, *Preprint* 173; 21 pp.

The paper presents the results of work carried out to study the influence of severe notches on the brittle-fracture behaviour of a vacuum-teemed forging steel of the following composition: carbon 0·25, silicon 0·28, manganese 0·61, phosphorus 0·01, sulphur 0·022, nickel 3·27, chromium 0·2, molybdenum 0·53, vanadium 0·08, copper 0·08, per cent.

The investigation was concerned primarily with investigation of the stresses and strains necessary to initiate brittle failure in a large, flat tensile specimen containing a pre-existing cleavage crack, such as are known to exist in large steel sections and structures (e.g., in the form of weld cracks, inclusions, flakes, shrinkage cracks, forging bursts, etc.). These crack-notch experiments were supplemented by tensile tests on notched and un-notched small cylindrical specimens, as well as by conventional and low-blow Charpy V-notch impact tests. To minimise the hydrogen content, all the specimens were baked at 212°F. (100°C.) before testing.

The results show that the fracture-strength of

the sharply notched steel was highly dependent upon temperature and exhibited, over a narrow temperature range, a pronounced transition from a value well above to well below the normally measured yield strength. This transition was associated with a change in the mode of fracture initiation from a ductile shear tear to a brittle cleavage fracture. For the particular crack-notch condition studied, brittle fractures were obtained within the stress and temperature ranges to which this type of steel is subjected in practice, and the results are therefore indicative of the possible causes of service failures.

The critical stresses and temperatures at which brittle fractures initiated in the presence of pre-existing cracks were found to be dependent upon geometric factors. Increasing the crack size in a constant section decreased the fracture strength in proportion to the inverse square root of the crack length. Increasing the total size (maintaining a constant ratio between crack length and section size) would be expected to produce similar results in accordance with predicted behaviour. It is therefore emphasised that the practical application of such fracture-strength data requires consideration of both crack- and section-size effects.

The ductile-to-brittle-transition behaviour exhibited by the specimens subjected to the crack-notch test is compared with that determined by means of other tests. The transition temperature as established by either the 'low-blow' or 'drop-weight' techniques is considered to provide satisfactory indication of the temperature range in which the change in the mode of fracture initiation, and the associated sharp decrease in fracture strength, can be expected to occur.

Development of Non-Embrittling Cadmium-Plating Processes for High-Strength Steels

P. N. VLANNES and S. W. STRAUSS: 'Progress toward the Development of a Non-embrittling Cadmium Electroplating Process. II. Use of Aliphatic Amino Acids as Complexing Agents in Aqueous Baths.'

Plating, 1959, vol. 46, Sept., pp. 1046–51.

Non-cyanide aqueous plating solutions containing additions of β-alanine or an α-amino acid (glycine, n-butyric, isobutyric, n-caproic or isocaproic) were studied, over a wide pH range, to determine their embrittling effects on ultra-high-strength steel. Plating characteristics were evaluated by means of the Hull-cell and by throwing-power tests; embrittling characteristics were assessed on the basis of delayed-fracture tensile tests on notched bars of nickel-chromium-molybdenum steel (A.I.S.I. 4340) heat-treated to a strength of approximately 285,000 p.s.i. (127 t.s.i.; 200 kg./mm.²).

The results of the delayed-fracture tests indicated that plating from an ammoniacal cadmium solution containing salts of amino acids resulted in markedly lower susceptibility to hydrogen embrittlement than plating from the standard cyanide bath.

(For abstract of Part I of the series see *Nickel Bulletin*, 1959, vol. 32, No. 8, p. 259.)

Influence of Hot-Cold Working on the Properties of High-Strength Steels

J. C. SHYNE, V. F. ZACKAY and D. J. SCHMATZ: 'The Strength of Martensite Formed from Cold-Worked Austenite.'

Amer. Soc. Metals, 1959, *Preprint* 163; 16 pp.

Increases in the strength of martensitic steels in recent years can be attributed primarily to the use of lower tempering temperatures and, to a certain extent, have been obtained at the expense of ductility and toughness. The effects of modifying composition and heat-treatment are likely to be limited, and efforts are being made to discover other ways of increasing the strength of steels. Unusually high strength has been variously reported to have been obtained in steels by hot-cold working of metastable austenite prior to quenching to room temperature. The present paper describes a study of the influence of such thermal-mechanical treatment on the microstructure of five experimental steels.

Because of the time necessary to hot-cold work the metastable austenite, the composition of the steels studied (see table below) was designed to ensure the presence of an austenitic 'bay' in the TTT diagram. The steels, induction-melted in air or *in vacuo*, were forged to bar and normalised. Samples were then austenitised at 1600°F. (870°C.) and cooled in air to a deformation temperature below the recrystallisation temperature of the austenite, but above the M_s temperature. The deformation temperature was confined to the temperature range of the metastable 'bay', i.e., between 800° and 1050°F. (425° and 565°C.). Immediately after the metastable austenite had been plastically deformed to the desired extent (0–93 per cent. reduction-in-area), the samples were quenched in oil. Undeformed specimens were also cooled from 1600°F. (870°C.) to room temperature by this same uninterrupted cooling path. Deformation was effected by multi-pass rolling to round bar stock. The influence of this treatment was studied in relation to tensile properties, microstructure, retained austenite and transformation kinetics.

C %	Cr %	Ni %	Mn %	Si %	Mo %	V %
0·31	2·21	1·03	1·15	1·59	0·31	0·31
0·41	2·23	1·07	1·17	1·64	0·34	0·32
0·47	2·27	1·05	1·10	1·46	0·34	0·32
0·48	2·94	1·65	0·75	1·53	0·42	—
0·63	2·91	1·57	0·75	1·54	0·43	—

Hot-cold working the austenite was found greatly to accelerate the kinetics of transformation to non-martensitic decomposition products, and the martensite formed possessed greater tensile strength than when hardened conventionally. Increases as high as 145,000 p.s.i. (64·5 t.s.i.; 102 kg./mm.²)

in the ultimate tensile strength were observed. The greatest increase in tensile strength was associated with the largest degree of austenite deformation. Plastic deformation of the austenite resulted also in a refinement of the martensite; the decrease in martensite plate size being roughly proportional to the degree of austenite deformation. The simultaneous increase in strength and decrease in martensite size can be correlated in a manner consistent with current theories of yield and fracture.

The amount of retained austenite was not greatly influenced by hot-cold working prior to quenching, nor did the observed minor changes in retained austenite content affect the tensile properties to any considerable degree.

Tempering the martensite transformed from deformed austenite resulted in a great increase in its yield strength.

Influence of Lead Additions on the Machinability of Low-Alloy Nickel-Chromium-Molybdenum High-Strength Steels

N. ZLATIN and J. V. GOULD: 'The Effect of a Lead Additive on the Machinability of Alloy Steels.'

Trans. Amer. Soc. Mechanical Engineers, 1959, vol. 81, May, pp. 131-5; disc., pp. 136-8.

During the past 10 years additives such as sulphur, selenium or lead have been used to improve the machinability of medium-carbon low-alloy steels. These free-machining grades have, however, been used principally at hardness levels below 300 Brinell, and the trend towards higher-strength steels with hardnesses in the region of 400 Brinell has complicated the machining problems involved. A solution to these problems has been sought in the use of lead additives.

The investigation described by the authors was carried out to determine at what hardness levels such lead additions would be most effective. Turning tests were carried out, using carbide tools, on leaded and un-leaded test bars of two low-alloy steels: a chromium-molybdenum steel (A.I.S.I. 4147H) and a nickel-chromium-molybdenum steel (A.I.S.I. 4340H). Both the leaded and plain specimens were obtained from the same heat of steel and were heat-treated to various levels of strength. Machinability was assessed using the following criteria (to which the data reported relate): tool life; coefficient of friction between the sliding chip and the tool; tool forces and power consumption; and surface finish. The conclusions drawn on the basis of these criteria are quoted below.

'(1) The use of lead additive in medium-carbon alloy steels will result in appreciable increases in tool life for those steels having a hardness of R_c 30 or less. The advantage of the lead additive will decrease as the hardness of the steel increases beyond R_c 30. At some point in the hardness range of R_c 35 to R_c 40 or higher, the presence of the lead additive may be detrimental to tool life.

'(2) Appreciable improvement in surface finish is obtainable on medium-carbon alloy steels, regardless of the hardness of the steel.

'(3) The addition of lead to medium-carbon steels at various hardness levels will result in a reduction of 15 to 25 per cent. in power required in machining.'

Determination of Magnetic Phases Present in Steels

See abstract on p. 343.

Joining of Steels to Dissimilar Materials

See abstract on p. 344.

Hot Cracking in Low-Alloy-Steel Welds

P. W. JONES: 'An Investigation of Hot Cracking in Low-Alloy-Steel Welds.'

British Welding Jnl., 1959, vol. 6, June, pp. 282-90. *Part I* of *Report F.M.* 3/128/58 issued to members of the British Welding Research Association in Dec. 1958.

Cracking in the heat-affected zone of welded low-alloy high-strength steels has been the subject of much study. Welds in such steels are, however, themselves liable to cracking, and, since either form of cracking might be equally deleterious, an investigation of weld-metal cracking was undertaken by the British Welding Research Association. It was desired to determine whether weld-metal cracking could still occur under conditions precluding cracking in the heat-affected zone, and to provide information that would lead to specification of a test or to determination of the precautions necessary for avoidance of such failure. The present paper is concerned with hot cracking: a parallel investigation of cold cracking (fissuring) is reported in the paper to which reference is made in the following abstract.

Of the five types of test evaluated with the aim of finding a suitable method of assessing the susceptibility of hot-cracking, the Murex test, the only one to give consistent results, was adopted for use in the study described. (In the Murex hot-cracking test a controlled relative motion is maintained between two test plates during deposition of a fillet weld under standard conditions. The severity of any resultant cracking is assessed by measuring the length of the crack.) Details are given of the electrodes and test conditions employed in the investigation. Tests were made both on low-alloy and mild steel plates, the low-alloy steels comprising the following types: molybdenum-boron, manganese-nickel-chromium-molybdenum, manganese-molybdenum, 2 per cent. nickel, nickel-chromium-molybdenum, and nickel-chromium-molybdenum-vanadium.

Susceptibility to weld-metal cracking was studied in relation to the following factors:

(1) The influence of the carbon, nickel, copper, chromium, cerium and aluminium contents of the weld.

The alloying element was introduced into the weld either *via* the basis metal (to which the element

was added by a diffusion heat-treatment, the weld metal being enriched during welding by the normal process of dilution) or *via* a wire of the desired size which was placed at the root of the fillet before welding. To avoid interference by other elements the weld tests were carried out using mild-steel basis plate.

(2) Variation in hot-cracking susceptibility as a function of the composition of the low-alloy-steel basis metals studied.

(3) Effects of preheat.

(4) Effects of weld size.

The conclusions drawn with respect to the influence of the composition of the steel are quoted below:

'1. In view of the beneficial effect of a high manganese/sulphur ratio, there are advantages to be gained by keeping the sulphur content as low as possible. This applies particularly if, for any reason, it is desired to limit the manganese content. A high manganese/sulphur ratio permits the presence of the maximum amount of those useful alloying elements which increase hot cracking, without exceeding a given level of hot-cracking susceptibility.

'2. Manganese, in amounts of the order of 1 per cent., exercises a beneficial effect on the resistance to hot cracking of low-alloy steels.

'3. Carbon increases hot cracking, and when steels of relatively high carbon content (0·3 per cent. and over) are welded under restraint, it appears advisable to limit the dilution to ensure that the carbon content of the weld metal does not exceed about 0·15 per cent.

'4. Nickel increases hot cracking, but relatively large amounts (about 1 per cent. in the weld metal) may be tolerated without serious cracking, provided that a class 6 electrode, giving a high manganese/sulphur ratio in the weld metal, is chosen. Steels containing less than 2 per cent. nickel would not be expected to give rise to hot cracking due to this element, provided that reasonable precautions were taken.

'5. Insufficient tests have been carried out to confirm the inference that chromium is beneficial to hot cracking, and may possibly counteract the harmful effect of nickel.

'6. An indication has been obtained that copper, in amounts of the order of 1 per cent. in the weld metal, increases hot cracking. This large amount is not likely to be found in low-alloy-steel weld metals.

'7. An indication has been obtained that Mischmetall (50 per cent. cerium) may reduce hot cracking.'

It is considered difficult to specify an effective hot-cracking test, but the following limits are tentatively prescribed for elements which promote or mitigate hot cracking in the weld metal: sulphur 0·035 max., manganese 0·8 min., nickel 1 max., carbon 0·15 max., per cent.; manganese/sulphur ratio 35 min.

The results also show that pre-heating at temperatures up to 250°C. does not materially influence the severity of hot-cracking. In consequence, where pre-heating is specified as a means of preventing cracking in the heat-affected zone, the treatment may be applied without fear of occurrence of hot cracking. Small variations in weld size were shown to have no effect on hot-cracking in the Murex test.

See also:

Cold Cracking in Low-Alloy-Steel Welds

T. E. M. JONES: 'Cracking of Low-Alloy-Steel Weld Metal.'

British Welding Jnl., 1959, vol. 6, July, pp. 315–23. *Part II* of *Report F.M.* 3/128/58 issued to members of the British Welding Research Association in Dec. 1958.

The investigation was carried out with the aim of (1) defining the conditions which would ensure freedom not only from fissuring (cold cracking) in the weld metal, but also from cracking in the heat-affected zone, or (2) developing a test suitable for evaluating the tendency of a given combination of electrode and basis metal to produce cracking in the weld metal.

Six low-alloy steels were selected for study as basis metals: manganese-nickel-chromium-molybdenum, carbon - manganese, and two grades of manganese-molybdenum and chromium-molybdenum steel. A rutile-coated electrode (*B.S.* 1719, *Class E* 3) was used in most of the tests.

In the initial stages of the programme a 'long-plate' test was devised, by which the cooling rate of a series of bead welds deposited on a plate of standard thickness was varied uninterruptedly throughout the cooling cycle, as in a normal weld, using different electrode sizes to vary the heat input to the plate. Each weld was then sectioned for metallographic examination to determine, as revealed by an electrolytic etching technique, the number and orientation of the fissures present. This test is claimed not only to determine susceptibility to weld-metal cold cracking, but also to reveal heat-affected zone or underbead cracking.

Tests were made to determine: (1) the influence of bead size on the cooling rate and on the incidence and orientation of fissures; (2) the influence, on susceptibility to fissuring, of plate thickness and the presence of nickel and chromium in the weld metal; (3) the effects of reducing weld restraint; (4) the effects of reducing the amount of hydrogen available to the weld; (5) the effects of varying the alloying contents of chromium-molybdenum-steel electrodes; (6) the relation of fissuring to underbead cracking.

From the data presented the author draws the following conclusions:

'(*a*) Cold cracking or fissuring in weld metal depends on the rate of cooling, whether influenced by heat input to the weld, or by increased mass of metal in the weld vicinity. Slower cooling reduces fissuring.

'(*b*) The defect is confined to weld deposits of high potential hydrogen content. This was shown by a few tests in which the quantity of hydrogen available to the weld metal was much reduced, by the use of either the self-adjusting arc process (where the atmosphere consists of inert gas) or manual electrodes with a low hydrogen coating.

'(*c*) In the presence of hydrogen, such as is present from a normal rutile-coated electrode, chromium and nickel derived from the steel plate increase fissuring.

'(*d*) In tests in which the weld restraint was reduced, far fewer fissures were found than in corresponding welds with normal restraint.

'(*e*) Orientation of fissures changes, with weld bead size, from predominantly transverse in small welds to random in large welds.

'(*f*) In some tests a reduction in the amount of heat-affected-zone cracking was accompanied by an increase of fissuring.

'It is not always possible to assume that conditions which give safety from heat-affected zone cracking will not lead to the formation of fissures. However, reduction of the amount of hydrogen available in the arc atmosphere will reduce the likelihood of either defect occurring. Since contraction stresses affect fissuring, reduction of the cooling rate by some means other than the use of large welds will minimise both defects.'

HEAT- AND CORROSION-RESISTING MATERIALS

Stress-Rupture Testing of Steel: British Standard

BRITISH STANDARDS INSTN.: 'Mechanical Testing of Steel at Elevated Temperatures. Part 2. Stress Rupture.'

B.S. 3082: *Part* 2: 1959; 9 pp.

This British Standard, authorised by the Iron and Steel Industry Standards Committee, is one of a series dealing with the mechanical testing of steel at elevated temperatures, which, it is hoped, will help to facilitate comparison of different steels and unify the methods by which high-temperature data are obtained. (For abstract of *Part 1* see *Nickel Bulletin*, 1959, vol. 32, No. 5, p. 138.) The procedures covered are for determination of the time-to-rupture of a steel specimen subjected to a constant tensile stress at an elevated temperature, and are applicable to tests on single-specimen testing machines and to each test on multi-specimen testing machines.

Definitions and symbols used in stress-rupture testing are defined and requirements are specified with respect to: test piece; determination of elongation; heating apparatus and permissible deviations from the specified temperature for temperatures up to 1000°C.; measurement of temperature; heating of test piece and soaking period; testing machine; loading; temperature and stress-rupture readings; presentation of results; extrapolation.

Ferrous and Non-Ferrous Bourdon Tubing: British Standard

BRITISH STANDARDS INSTN.: 'Ferrous and Non-Ferrous Bourdon Tubing.'

B.S. 3127: 1959; 25 pp.

The Standard, which covers ferrous and non-ferrous tubing supplied in straight lengths in a suitable condition for the fabrication of pressure-responsive components of gauges and instruments operating on the bourdon principle, is in ten sections. Section one lays down general requirements with respect to manufacture, condition, heat-treatment, freedom from defects, straightness, sampling for tests, hardness test, etc. In section ten, dimensions and tolerances are specified for a series of standard sections for oval tubing: requirements for other sections are the subject of agreement between purchaser and manufacturer. The intermediate sections, covering eight tubing materials, lay down limits of composition and the condition (and related hardness) in which the tubing is to be supplied. Appendix A outlines the mercurous nitrate test to which brass tubing is to be subjected; Appendix B indicates the mechanical properties which the tubing may be expected to exhibit in the condition as supplied to the instrument manufacturer; Appendix C contains metric conversion tables. The tubing materials to which the Standard relates, and the limits of composition of the nickel-containing materials, are noted below.

Beryllium-Copper Tubing: beryllium 1·7–1·9, cobalt and/or nickel 0·05–0·4, total impurities 0·5 max., per cent., remainder copper.

Brass Tubing

Nickel-Copper-Aluminium Alloy Tubing: aluminium 2–4, titanium 0·25–1·0, copper 27–33, carbon 0·25 max., silicon 1·0 max., manganese 1·5 max., sulphur 0·01 max., iron 2·0 max., total impurities 0·2 max., per cent., nickel (including cobalt) remainder, but not less than 63 per cent.

Phosphor-bronze tubing

Chromium-molybdenum alloy-steel tubing

Stainless-steel tubing: carbon 0·08 max., silicon 1·0 max., manganese 2·0 max., chromium 16–18, nickel 10–14, molybdenum 2–3, phosphorus 0·04 max., sulphur 0·03 max., per cent.

Carbon-steel tubing

Iron-base, nickel, chromium, titanium (precipitation-hardening) alloy tubing: carbon 0·06 max., silicon 1·0 max., manganese 0·8 max., chromium 4·9–5·5, nickel (including cobalt) 41–43, phosphorus 0·04 max., sulphur 0·04 max., aluminium 0·3–0·8, titanium 2·1–2·6, total impurities 0·2 max., per cent., remainder iron.

Evaluation of Materials for High-Temperature Service

J. J. HARWOOD and N. E. PROMISEL: 'Approaches to the Problems of High Temperatures: A Comparative Materials Evaluation.'
Paper to *Third Plansee Seminar*, July 1958.
Plansee Proceedings, 1958, pp. 223–48.
Published by *Metallwerk Plansee A.G.*, 1959.

The authors introduce their review by drawing attention to the four categories which cover all the more important sources of high temperatures: power plants, aerodynamic heating, equipment operation, reactions (chemical, nuclear, thermonuclear). This introductory section is followed by a general discussion of the factors which govern selection of materials for high-temperature service and by references to techniques employed to lower, where feasible, the elevated temperatures involved in certain industrial applications (cooling, insulation, control emissivity, and ablation).

The main section of the review is concerned with an evaluation of materials for use at temperatures up to 650°C. (aluminium and titanium alloys, chromium-nickel corrosion-resisting steels, hot-work die steels, 'Inconel X' and 'Unitemp 212'), at temperatures in the range 650°-1000°C. (nickel-, cobalt- and iron-base 'superalloys'), and at temperatures above 1000°C. (refractory metals and molybdenum-base alloys). The textual information is supplemented by numerous curves illustrating the variation, with temperature, of the strength properties of the different groups of material. In a brief final section the author discusses the potential of cermets and graphite in relation to high-temperature applications.

Development of Controlled-Transformation Stainless Steels

K. J. IRVINE, D. T. LLEWELLYN and F. B. PICKERING: 'Controlled-Transformation Stainless Steels.'
Jnl. Iron and Steel Inst., 1959, vol. 192, July, pp. 218–38.

The demands of the aircraft industry for a structural material which would exhibit satisfactory properties at service temperatures up to the region of 400°C. have focussed attention on the advantages offered by high-strength stainless steels in this connection. To obtain the required strength/weight ratio the steel would have to possess a tensile strength of at least 70 t.s.i. (110 kg./mm.^{2}; 157,000 p.s.i.). Such strength levels are easily obtained in the available non-transformable and transformable standard grades of stainless steels, but their major disadvantage lies in the difficulties involved in fabrication. In the cold-worked condition the non-transformable ferritic or austenitic steels possess little ductility, and at elevated temperatures the effects of cold-deformation are nullified. The transformable martensitic steels can be easily fabricated in the fully-softened condition, but subsequent heat-treatment can result in serious scaling and distortion. The answer to the problem, therefore, lay in the development of controlled-transformation stainless steels, in which the M_s point is adjusted to just below room-temperature. The steel is austenitic when cooled to room temperature and can therefore be easily fabricated; refrigeration treatment below the M_s point or heat-treatment at 700°C. will then cause transformation, due, in the latter case, to precipitation of alloy carbides which raise the martensite transformation range. Addition of precipitation-hardening further increases the strength of the martensitic structure.

Developments along these lines have in recent years resulted in the production of high-strength stainless steels deriving their properties from a simple transformation mechanism or from a combination of transformation and ageing mechanisms, e.g., '17-4 PH', '17-7 PH', '15-7 PH Mo', 'AM-350', 'Stainless W', 'F.V. 520'. In view of the extremely attractive properties of such steels and the possibility of their becoming standard engineering structural materials, it was thought desirable to review the types of steel obtainable and to explain the mechanisms involved. The scope of this comprehensive review is indicated below, under the heads used by the authors.

I. Background to Controlled-Transformation Stainless Steels.

1. *Essential Features*

The main compositional and structural requirements of controlled-transformation steels are reviewed. Two typical compositions are selected as representative of such steels: carbon 0·1, chromium 17, nickel 4, molybdenum 2, per cent.; carbon 0·1, chromium 17, nickel 4, manganese 3, per cent. Their main features are outlined in relation to the factors indicated in the sections below, discussion of which is supplemented by numerous data.

2. *Martensitic Transformation Range.*

3. *Influence of the Solution-Treatment Temperature on the Martensitic Transformation Range*

It is emphasised that the solution-treatment temperature provides a means of controlling the amount of carbide taken into solution and, hence, of controlling the effective composition, both with regard to alloying elements and carbon content.

4. *Effect of Refrigeration*

5. *Effect of Primary Tempering in Raising the M_s Point*

6. *Effect of Secondary Tempering on the Strength of the Martensitic Structure*

7. *Use of Cold Work to Produce Transformation*

8. *The Precipitation-Hardening Mechanism*

Data are included illustrating the effects, on the strength of the molybdenum-containing steel, of age-hardening additions of copper.

9. *Microstructures*

Photomicrographs illustrating the microstructures of the two steels in various conditions of heat-treatment are discussed.

10. *Mechanical Properties*

The discussion is supplemented by data indicating the properties resulting from various heat-treatments, the variation of these properties with the amount of martensite present, the effect of cold work in producing transformation, and the effect of tempering cold-rolled sheet. The influence of age-hardening is exemplified by reference to data on the properties of the copper-bearing steel.

II. Experimental Work on Controlled-Transformation Steels.

The work described in the first two sections below was carried out on a basis composition consisting of carbon 0·1, chromium 17, nickel 4, per cent. The factors governing the choice of this composition are discussed.

1. *Effect of Alloying Elements on the* 17 *Cr–4 Ni Base*

The influence of the following alloying additions was determined, with the aim of establishing those which would ensure optimum control over ferrite content and/or the position of the martensite transformation range: nitrogen, carbon, nickel, cobalt, copper, manganese, tungsten, silicon, molybdenum, chromium, vanadium, aluminium.

The authors also discuss the results of experiments to determine the influence, on the transformation characteristics of the 17 Cr–4 Ni basis composition, of alloying additions of manganese, silicon, molybdenum, tungsten, vanadium, aluminium, copper and cobalt.

2. *Secondary Hardening*

The age-hardening effects of copper, cobalt, aluminium, titanium and niobium are described in relation to 17–4–Mo and 17–4–Mn steels.

3. *Development of High-Strength Steels*

This section describes work on the development of optimum compositions from the 17–4–Mo and 17–4–Mn bases. Data are tabulated on the properties of the six steels developed. Very high levels of strength were obtained with the following age-hardenable steels: carbon 0·06, chromium 16·4, nickel 5·14, manganese 2·09, molybdenum 1·51, aluminium 0·88, cobalt 2·03, per cent.; carbon 0·07, chromium 17, nickel 3·5, manganese 2, molybdenum 2, copper 1·2, cobalt 2, per cent.

III. Conclusions.

The authors' main conclusions are quoted below.

'Satisfactory controlled-transformation steels can be developed from the 17 Cr–4 Ni basis composition by making single additions of manganese, molybdenum, cobalt, silicon, tungsten, and vanadium. These steels all have 0·05–0·10 per cent. carbon, but because the structure is essentially martensitic, higher strengths can be obtained by raising the carbon content, although this will be at some sacrifice of corrosion-resistance, ductility, and welding characteristics.

'It has been found that certain alloying elements promote a strong age-hardening reaction, and copper, aluminium, cobalt, titanium and niobium have been studied. The ageing reaction leads to a considerable increase in the strength of the martensitic structure, and it is found that when one of these compositions is tested, the final properties are far less dependent on a complete transformation to martensite. Consequently, a controlled-transformation ageing steel would appear to offer the most promise for commercial development.

'The essential requirements for one of these controlled-transformation ageing steels are:

(i) A low carbon content.

(ii) Sufficient chromium for adequate corrosion-resistance.

(iii) Nickel to obtain an essentially austenitic structure with a controlled transformation.

(iv) Manganese to provide control over transformation temperature without affecting the microstructure.

(v) Molybdenum to provide tempering resistance and improved high-temperature strength.

(vi) Copper, cobalt or aluminium to provide an ageing reaction; improved properties are obtained when two of these reactions are combined.

(vii) Titanium or niobium for maximum resistance to weld decay.'

Dispersion-Hardening of Nickel with Aluminium Oxide

W. M. SCHWARZKOPF and N. J. GRANT: 'Fine-Particle Strengthening for High-Temperature Use.'
Paper to *Third Plansee Seminar*, July 1958.
Plansee Proceedings, 1958, pp. 454–63; disc., pp. 463–5. Published by *Metallwerk Plansee A.G.*, 1959.

The successful application of dispersion-hardening to aluminium products (e.g., of SAP type) has encouraged many investigators to seek similar strengthening effects in more refractory systems. Not all metals can be treated in the same manner as aluminium, however, and other means had to be found of incorporating in the matrix material a fine dispersion of the second phase. Methods suggested include (*a*) direct mixing of the fine powders, (*b*) surface coatings of one constituent on the other, (*c*) a diffusion technique such as internal oxidation, (*d*) reaction of the powder constituents to produce a dispersion of an intermetallic phase insoluble in the matrix. Many of the refractory oxides possess properties which render them ideal for use as the finely-dispersed phase, but insoluble phases other than oxides have also been employed: examples are referred to in the literature survey which opens the present paper. The aim of the paper is to present data illustrating the effects of dispersion hardening on the high-temperature strength of various materials, and to compare the results obtained using different hardening procedures.

The influence of interparticle spacing is discussed and data indicating the stresses for 100-hour rupture

life at various temperatures are tabulated for copper, nickel and aluminium dispersion-hardened with Al_2O_3, for molybdenum hardened with ZrO_2, and for SAP.

The main part of the paper is concerned with a discussion of the relative efficacy of two dispersion-hardening techniques, as applied to the hardening of nickel with a dispersion of Al_2O_3. The Al_2O_3 was introduced (1) by mixing it with the nickel powders, or (2) by internal oxidation (i.e., by diffusing oxygen into fine powders of a dilute solid-solution of aluminium in nickel, such that the Al_2O_3 dispersion is produced *in situ*). The data on properties presented by the authors relate to materials hardened by internal oxidation; the comparative data on nickel hardened by mechanical mixing with Al_2O_3 is contained in a paper by CREMENS and GRANT (see *Proc. Amer. Soc. Testing Materials*, 1958, vol. 58, pp. 714–32; abstract in *Nickel Bulletin*, 1958, vol. 31, No. 8, pp. 229–30).

The diffusion method is stated to be applicable only to those systems in which the constituents exhibit appreciable differences in their affinity to oxygen. It is therefore not nearly as comprehensive as the direct method of mixing the component powders, but, nevertheless, the technique offers important advantages. Metal powders less than 1 micron in size are exceedingly expensive and difficult to handle. Furthermore, even though oxides such as Al_2O_3 can be obtained in sizes in the region of 0·02 micron, work carried out in the authors' laboratory indicates that these oxides are greatly agglomerated, and in this form contribute only a fraction of their potential high-temperature-strengthening effect to the composite metal/metal-oxide product. The internal-oxidation technique enables production of a much finer and more uniform dispersion with alloy powders as much as 20–40 microns in diameter. The pre-alloyed powders used in the present investigation, supplied by The International Nickel Company, Inc., were produced by first casting the alloys into a cylindrical shape and then, using a rotary file, rendering them into elongated chips having a cross-sectional diameter of roughly 80 microns and giving a diffusion path of half that distance. Data are presented illustrating the relationship between penetration depth and temperature of oxidation for nickel-chromium and nickel-aluminium alloys.

Thermal Stability of Precipitation-Hardened and Dispersion-Hardened Nickel-base Alloys

D. H. FEISEL and A. COCHARDT: 'Comparison of the Thermal Stability of Some Precipitation-Hardened and Dispersion-Hardened Nickel-base Alloys.'

Trans. Metallurgical Soc. Amer. Inst. Mechanical Engineers, 1959, vol. 215, Aug., pp. 608–13.

In a previous paper Cochardt discussed the factors which govern the process of over-ageing in precipitation-hardened high-temperature alloys and attempted an estimation of the time necessary to produce over-ageing of some of the hardener constituents most commonly used in nickel-base alloys (*ibid.*, 1957, vol. 209, pp. 434–7; see abstract in *Nickel Bulletin*, 1957, vol. 30, No. 7–8, p. 130). His conclusions indicated that alloys hardened with Ni_3Al, TiC or TiN are unstable at 1340°F. (725°C.), but that those hardened with alumina should be virtually immune to over-ageing. The investigation now recorded was carried out to determine, on the basis of experimental results, the validity of these conclusions.

Six alloys were prepared for testing (see table below). The compositions of the three wrought precipitation-hardening alloys were designed to ensure precipitation of 5 vol. per cent. of the respective hardening phase at 1350°F. (730°C.), the temperature selected to illustrate the effect of ageing time on structural stability. Specimens of these alloys were solution-treated for 24 hours at 2100°F. (1150°C.) in an argon atmosphere and water quenched. The remaining three alloys, produced by extrusion techniques, were dispersion-hardened with alumina particles. The structural stability of all six alloys was assessed on the basis of hardness measurements and the results of metallographic examination.

Precipitation-Hardened and Dispersion-Hardened Alloys

Al %	Ti %	C %	Cr %	Al_2O_3 %	Ni %
6·03	—	—	—	—	balance
—	7·31	—	—	—	balance
—	8·75	0·11	—	—	balance
—	—	—	—	5·81	balance
3·31	—	—	—	4·40	balance
3·35	—	—	16·35	3·94	balance

The behaviour of the nickel-aluminium, nickel-titanium and nickel-titanium-carbon alloys was found to be typical of that of precipitation-hardened alloys in general: initially the hardness increased rapidly with time (due to precipitation from the supersaturated solid solution), reached a maximum, then decreased with time as the process of over-ageing occurred. The alumina dispersion-hardened materials, however, behaved quite differently: no significant change in hardness from that of the as-extruded value occurred within 512 hours, indicating that the factors contributing to their high hardness (mainly the alumina dispersions, but possibly also the extremely fine grain sizes) were not greatly affected by time at this temperature.

Data presented on the effect of temperature on the hardness of these materials, for a constant annealing time of 16 hours, show that at 1350°F. (730°C.) the precipitation-hardened alloys were well over-aged after this time, and therefore did not show much change in hardness at higher temperatures. In contrast, the loss in hardness suffered by the dispersion-hardened materials was insignificant up to a temperature of approximately 2000°F. (1095°C.). Metallographic examination of the microstructure of the

dispersion-hardened alloys after 16 hours at 2150°F. (1175°C.) revealed a very slight but definite enlargement of the alumina particles, and since, within the limits investigated, their extremely fine grain size appeared unaffected, the loss in hardness suffered at temperatures above 2000°F. is considered to result from the growth of the dispersed alumina phase.

Total Hemispherical Emissivity of Nickel/Titanium-Carbide Cermets

W. R. WADE and F. W. CASEY: 'Measurements of Total Hemispherical Emissivity of Several Stably Oxidised Nickel-Titanium Carbide Cemented Hard Metals from 600°F. to 1600°F.'

Nat. Aeronautics and Space Administration, Memo. 5-13-59*L*, June 1959 ; 31 pp.

Tests on nickel/titanium-carbide cermets have indicated that they offer promise as materials of construction for the leading edges and nose cones of high-speed aircraft. The present investigation was initiated to determine, over a temperature range of 600°-1600°F. (315°-870°C.), the total hemispherical emissivity of representative materials in the as-received and as-polished conditions, and after subjection to oxidising treatments in each of the two conditions. The cermets studied contained 10, 20, 30, 33·3 or 40 per cent. of nickel, remainder titanium carbide and niobium carbide (and, in one case, also molybdenum, chromium and aluminium).

The results indicated that the values obtained by oxidation of the materials in the as-received condition are somewhat higher than those obtained after preliminary polishing: for the stably-oxidised as-received test specimens emissivity values ranged from 0·9 to 0·94 at 600°F. (315°C.) and from 0·88 to 0·92 at 1600°F. (870°C.); for those which were brightly polished prior to oxidation the values ranged from 0·82 to 0·89 at 600°F. and from 0·85 to 0·87 at 1600°F. Although emissivity may vary as a result of differences in the surface finish of the as-received materials, it is considered unlikely that the variation will exceed 2 per cent. for any specific material.

Data derived from tests on cermets containing only nickel, titanium carbide and niobium carbide suggest that the emissivity of the stably-oxidised surface increases with increase in nickel content, but the differences in the emissivity of the materials tested were so slight as to be inconclusive. Stable oxide coatings with the desired physical properties could not be produced on materials which, instead of nickel, contained cobalt as the metal phase, or which had a high molybdenum content.

Hot-Hardness of Nickel-Chromium Alloys

K. A. OSIPOV and E. M. MIROSHKINA: 'Investigation of Alloys of the System Nickel-Chromium by Hot-Hardness Tests.'

Trudy Inst. Met. im. A.A. Baikova, 1958, No. 3, pp. 160–4.

The hot-hardness of nickel-chromium alloys containing chromium 5·28, 7·63, 9·59 or 23·82 per cent. was determined at temperatures in the range 25°-1100°C., and the results obtained were correlated with data on creep behaviour. For details of the authors' findings see *Chemical Abstracts*, 1959, vol. 53, p. 13015.

Fatigue, Creep and Rupture Properties of 'Udimet 500', 'Hastelloy R-235' and 'GMR-235'

F. H. VITOVEC: 'Fatigue, Creep and Rupture Properties of the Alloys 'Udimet 500', 'Hastelloy R-235', and 'GMR-235'.'

Wright Air Development Center, Tech. Report 58-340, Oct. 1958; 63 pp.

The investigation described represents a further phase in a test programme designed to evaluate the static and fatigue properties of notched and unnotched specimens of commercial heat-resisting alloys. 'Udimet 500' is a nickel-cobalt-chromium-molybdenum-base alloy, and 'Hastelloy R-235' and 'GMR-235' are of nickel-chromium-iron-molybdenum base. (For particulars of alloys previously evaluated see abstract in *Nickel Bulletin*, 1957, vol. 30, No. 7-8, p. 128.)

Fatigue, rupture and creep data obtained from axial-stress tests, at 1200° and 1650°F. (650° and 900°C.), involving various combinations of alternating and mean stress, are presented in S-N curves and stress-range diagrams illustrating the effects, on fatigue life and creep behaviour, of stress concentration, temperature, the alternating-to-mean stress ratio, and stress magnitude.

Although the elevated-temperature strength of the three alloys was higher than that of previously investigated materials, the general mechanical behaviour was found to be similar. The author, however, makes specific reference to the following points:

(1) 'Hastelloy R-235' heat-treated for high strength at 1650°F. (900°C.) was, at 1200°F. (650°C.), in a notch-sensitive state, indicating that the heat-treatment must be suitably adjusted for optimum properties at service temperatures.

(2) The sequence in which the loads were applied to 'Hastelloy R-235' and 'GMR-235' had a significant effect on the strength of the two alloys. When the static load for small alternating-to-mean stress ratios was applied before the alternating stress, failure occurred on application of the alternating load. When the static load was applied after or during application of the alternating load, the specimens sustained the full loads for a significant time.

Creep-Resistance of Nickel-Copper Alloys

K. A. OSIPOV, E. M. MIROSHKINA and A. N. SOTNICHENKO: 'Creep-Resistance of Alloys of the System Nickel-Copper.'

Trudy Inst. Met. im. A.A. Baikova, 1958, No. 3, pp. 152–9.

Alloys containing from 19·34 to 89·88 per cent. of copper, remainder nickel, were annealed for 60

hours at 950°C. and tested to determine the relationship between resistance to plastic deformation and composition, temperature and time of deformation. The results of the investigation are noted in *Chemical Abstracts*, 1959, vol. 53, p. 13016.

High-Temperature Properties of Nickel- and Cobalt-base Alloys Melted and Cast in Air and *in Vacuo*

M. J. STUTZMAN and J. W. CUNNINGHAM: 'The Effects of Melting and Casting Procedures on the Elevated-Temperature Properties of Nickel and Cobalt-base Alloys.'

Trans. Metallurgical Soc. Amer. Inst. Mechanical Engineers, 1959, vol. 215, Aug., pp. 637–47.

The experiments described were carried out on 'Stellite 31', 'He 1049', 'Utimet 500' and 'Guy Alloy'. The information contained in the paper is closely cognate with that presented by STUTZMAN in *Wright Air Development Center Report* 57-678, an extended abstract of which appeared in *Nickel Bulletin*, 1959, vol. 32, No. 1, pp. 17–18.

Mechanical Properties of Air-Induction- and Vacuum-Arc-Melted Heats of 'W-545' Alloy

J. BULINA and J. T. BROWN: 'Evaluation of Properties Obtained from an Air-Induction and Vacuum-Arc Melted High-Temperature Alloy.'

Trans. Metallurgical Soc. Amer. Inst. Mechanical Engineers, 1959, vol. 215, Aug., pp. 571–7.

The investigation described was carried out to determine whether the mechanical properties of high-temperature alloys produced by vacuum cold-hearth arc-melting are inherently superior to those of same types of alloy produced by air-melting techniques.

'W-545', a high-temperature alloy of the following nominal composition, was selected for study: nickel 26, chromium 13, molybdenum 1·5, titanium 2·8, boron 0·03, per cent., remainder iron. Four heats of the alloy were evaluated: two laboratory heats and two pilot-plant production heats. One heat of each of the laboratory and pilot-plant alloys was air-induction melted, and the others were consumable-electrode vacuum-arc melted using air-induction-melted electrodes. All the specimens were given the following heat-treatment: solution-treated for 3 hours at 2000°F. (1095°C.), water-quenched, aged for 20 hours at 1375°F. (745°C.), furnace-cooled to 1200°F. (650°C.), held for an additional 20 hours, and air-cooled. The four heats were evaluated on the basis of 0·2 per cent. yield strength and percentage elongation at room-temperature and 1200°F. (650°C.), and by stress-rupture tests at 1200° and 1300°F. (650° and 705°C.). Details are given of the method used to derive a quantitative assessment of the relative efficacy of the melting methods from the properties obtained.

On the basis of this assessment it was found that the laboratory and pilot-plant consumable-electrode vacuum-arc-melted heats were both 13 per cent. superior to the air-induction-melted heats. The relatively constant superiority of the properties exhibited by the vacuum-melted alloys (despite the large differences in ingot size, amount of reduction, grain size, and specimen directionality associated with the two heats) leads the authors to conclude that the properties resulting from this melting practice are quantitatively superior to those of air-melted grades.

Influence of Addition Elements on the Elastic Limit of Cobalt-Nickel-Chromium-base Alloys

H. NOHL, B. LUX and W. SIEGFRIED: 'Study of the Effects of Addition Elements on the Elastic Limit of Austenitic Steels (Alloys) at Room Temperature.'

Mémoires Scientifiques de la Revue de Métallurgie, 1959, vol. 56, June, pp. 19–29.

In the introduction to the paper the authors draw attention to the importance of the elastic limit, as one of the criteria determining the suitability of a material for use in certain gas-turbine applications. In this connection, and by way of illustrating the beneficial effects of heat-treatment on the values obtained, reference is made to a table listing the elastic limits of various heat-resisting nickel-cobalt-base alloys in the as-cast and heat-treated conditions.

The aim of the investigation now described was to determine the influence of small amounts of alloying additions on the elastic limit ($E_{0·2}$ and $E_{0·01}$, i.e., the stresses corresponding to an elongation of, respectively, 0·2 and 0·01 per cent.), rupture stress, elongation, reduction-in-area, and hardness of two alloys (similar to the high-temperature alloys 'X-50' and 'X-63') of the following compositions: cobalt 40, chromium 22·5, nickel 20, tungsten 12, iron 2·5, carbon 0·76, manganese 0·6, silicon 0·5, per cent.; cobalt 58, chromium 23, nickel 10, molybdenum 6, iron 1, carbon 0·4, per cent. The influence of the following additions was studied: niobium, aluminium, titanium, titanium + aluminium, cerium, copper ('X-50'); niobium, aluminium, titanium, titanium+aluminium, boron, copper, copper+boron, cerium, manganese, copper +manganese+aluminium ('X-63'). The amounts of alloying additions varied with the individual addition, but fell within the range 0·05–4·54 weight per cent. Specimens were tested both in the as-cast condition and after ageing for 24 hours at 800°C.

It is concluded from the data presented that the alloying additions apparently have no beneficial effect on the $E_{0·2}$ elastic limit of either alloy, but that certain of the additions (e.g., copper and cerium) increased the $E_{0·01}$ elastic limit of the 'X-63' alloy.

Phase Changes in 'Incoloy 901' and 'Inconel X' during Creep Testing

C. C. CLARK and J. S. IWANSKI: 'Phase Changes in Precipitation - Hardening Nickel - Chromium - Iron Alloys During Prolonged Heating.'

Trans. Metallurgical Soc. Amer. Inst. Mechanical Engineers, 1959, vol. 215, Aug., pp. 648–51.

The aim of the investigation reported was to study

the microstructural changes occurring in the age-hardening iron-nickel-chromium-molybdenum-base and nickel-chromium-iron-base alloys 'Incoloy 901' and 'Inconel X' during long-time creep testing.

The creep-rupture data presented for 'Incoloy 901' were derived from tests at 1000°, 1200° and 1350°F. (540°, 650° and 735°C.); those for 'Inconel X' were obtained at 1500°F. (815°C.). Changes in the microstructures of the alloys were determined by X-ray and electron diffraction, electron microscopy, and use of an electron-probe microanalyser.

Some of the changes observed were similar in both alloys. In the case of 'Inconel X', equilibrium structure was not attained even after 35,000 hours, during which time the principal age-hardening phase changed from face-centred-cubic to a hexagonal η phase. A similar transformation began in 'Incoloy 901' after 1500 hours at 1350°F. (735°C.) and would, it is estimated, be virtually complete after 10,000 hours. Possible mechanisms for this transformation are discussed.

Ageing Effects in 'Nichrome' and 'Nimonic' Alloys

Y. S. AVRAAMOV, L. N. BELYAKOV and B. G. LIVSHITS: 'Internal Friction Peaks in Nickel-Chromium-base Solid Solutions.'

Fiz. Metal. i Metalloved, 1958, vol. 6, pp. 116–21.

Changes in properties observed in nickel-chromium and nickel - chromium - titanium - aluminium alloys after tempering and quenching were investigated by experiments, carried out on 'Nichrome' and 'Nimonic' specimens, to determine internal-friction/temperature curves. The authors' findings are indicated in *Chemical Abstracts*, 1959, vol. 53, p. 13015.

K-State in 'Nichrome' and 'Nimonic' Alloys

B. G. LIVSHITS, S. S. IBRAGIMOV, Y. S. AVRAAMOV and Y. K. KONEV: 'The Theory of the Phase Transitions in 'Nichrome' and 'Nimonic'.'

Issledovaniya po Zharoproch. Splavam Akad. Nauk S.S.S.R. Inst. Met. im. A.A. Baikova, 1957, vol. 2, pp. 171–80.

The development of the *K*-state in 'Nichrome' and 'Nimonic' alloys heated in the range 200°–700°C. was studied by electron microscopy, micro-hardness measurements and determination of internal friction. The results of the investigation are noted in *Chemical Abstracts*. 1959, vol. 53, p. 12988.

Protection of Molybdenum from Oxidation: Classification of Coating Systems

R. I. JAFFEE: 'Protective Coating Systems for Molybdenum.'

Contained in *Fabrication of Molybdenum*, pp. 119–33. Published by *Amer. Soc. Metals*, 1959.

Work on the development of coatings which will confer on molybdenum the oxidation-resistance necessary for full advantage to be taken of its excellent strength at high-temperatures is still in its initial stages. That this is so is evident from the wide range of coating systems suggested for the purpose, few of which have progressed beyond laboratory development. This paper was written in an attempt to satisfy the need for a classification of, and an evaluation of the properties required in, these systems. The review is based on a *bibliography* of 34 items.

The coatings classified are noted below.

(1) *Silicide Coatings* applied by vapour deposition.

(2) *Al-Si-Cr Coatings* applied by spraying and sintering.

(3) *Nickel Coatings* applied by electrodeposition or cladding.

(4) *Chromium Coatings* applied by electrodeposition or by a cementation process.

(5) *Ni-Cr-Alloy Coatings* applied by:

- (*a*) diffusion after alternately plating the molybdenum with nickel and chromium;
- (*b*) cladding with 'Nichrome' and 'Inconel';
- (*c*) fusion of brazing alloys such as 'Colmonoy C' or 'Coast Metal 50'.

(6) *Frit-type Coatings*.

(7) *Al-Si Coatings* applied by hot-dipping or spraying.

(8) *Other Coatings* (e.g., electrophoretic coatings of Ni-Cr alloy, cementation coatings).

In each of these sections the method of application is briefly described and the advantages and limitations of the respective coating are outlined.

The second part of the paper is concerned with discussion of the characteristics required in protective coatings if the advantages offered by molybdenum at high temperatures are not to be diminished or nullified. The coatings should have no adverse effects on the strength, ductility, thermal expansion and thermal conductivity of the molybdenum, and oxidation-resistance is, of course, a primary requirement. In considering each of these factors, the author briefly evaluates the suitability of the various coatings.

Protective Coatings for Molybdenum Turbine Blades

M. A. LEVINSTEIN: 'The GE Molybdenum Bucket Story: Coating Development and Evaluation.'

Contained in *Fabrication of Molybdenum*, pp. 186–204. Published by *Amer. Soc. Metals*, 1959.

In this paper the author presents the results of work, carried out by the General Electric Company, on the development of protective coatings for molybdenum gas-turbine blades. The work comprised: (1) initial studies of coated panels under laboratory conditions, (2) evaluation of experimental coatings in engine tests at 1500°F. (815°C.), (3) engine tests, under steady-state and cyclic conditions, on a multi-layer clad system at 1800°F. (980°C.) and above. The paper has been previously abstracted from an abridged version, and reference to the preparation and testing of the four-layer coating which gave

the most promising results will be found in the relevant abstract in *Nickel Bulletin*, 1959, vol. 32, No. 9–10, p. 297.

The scope of the overall test programme (blade design and manufacture, the development of the 'hot-rod' test engine, and operational problems) is outlined in the following paper:

R. C. DOWNEY: 'The GE Molybdenum Bucket Story: Testing in the 'Hot-Rod' Engine.'
Ibid., pp. 174–85.

Forming of Molybdenum Sheet Roll-Clad with 'Inconel' or 'Nichrome V'

D. C. GOLDBERG: 'Forming of Clad Molybdenum.' Contained in *Fabrication of Molybdenum*, pp. 148–68. Published by *Amer. Soc. Metals*, 1959.

In relation to such applications as turbine blades, discs and vanes, the use of molybdenum sheet offers advantages as compared with forgings. In sheet form the high thermal conductivity of the metal can be utilised to better effect and its relatively high density is offset by the thinness of the component. Moreover, the problem of protecting the metal from oxidation at high temperatures is simplified by the possibility of cladding the sheet, during rolling, with an appropriately oxidation-resistant material. This paper reviews work carried out, by Westinghouse Electric Corporation, on the application of roll-cladding techniques to molybdenum.

The basic technique used involved welding a box of the cladding material so as to permit insertion of the molybdenum core at the open end. The thickness of the cladding material was selected to result in a cladding, on each side of the core, 5–10 per cent. of the thickness of the finished composite (the finished sheet would be about 50–60 mils thick). The cladding was cleaned, the molybdenum core inserted, and the unit was then vacuum-annealed at 2200°F. (1205°C.). After annealing, the box was flushed with helium or argon and the open end was welded to enclose the molybdenum core. The composite was then ready for rolling.

Research on cladding techniques has been previously described by the author in more detail in other papers (see, for example, *Trans. Amer. Soc. Metals*, 1956, vol. 48, p. 595; abstract in *Nickel Bulletin*, 1956, vol. 29, No. 3, p. 47), and in the work now described tests were conducted to evaluate the properties of molybdenum sheet clad with 'Inconel', 'Nichrome V', or nickel. In an attempt to minimise diffusion between the molybdenum and the cladding materials, tests were made also on clad sheet in which diffusion barriers of such materials as palladium, copper, nickel and 'Monel' were interposed between the cladding and the molybdenum.

The results of the following tests are presented in the paper:

(1) *Metallographic Examination of Hydrogen-Annealed Sections*

Photomicrographs of the molybdenum/'Incone interface reveal that a thin diffusion zone separates the work-hardened core from the larger-grained cladding.

(2) *Bend Tests at Various Temperatures*

The data obtained show that the 'Inconel'- and 'Nichrome'-clad sheet is, both in the as-rolled and in the annealed condition, satisfactorily ductile and can be bent to a large angle before fracture.

(3) *Tear Tests on 'Inconel'-Clad Sheet*

These tests (which were intended to determine the effects, on the strength of the bond, of variation in cladding technique and heat-treatment) revealed that slight variations in the canning technique had little or no influence on bond strength. Annealing for 50 hours at 1600°F. (870°C.) increased the workability of the material; annealing for longer times had detrimental results.

(4) *Oxidation Tests involving Exposure in Still Air at 1700°, 1900° and 2000°F. (925°, 1035° and 1095°C.) and Cyclic Tests*

Most of the coatings failed near the edges or corners. The 'Inconel' cladding exhibited the highest resistance to continuous oxidation.

(5) *Ballistic Impact Tests at Elevated Temperatures*

The results of these tests are considered satisfactory.

(6) *Engine Testing*

Various components (including after-burner nozzle leaves and nozzle diaphragms) were fabricated from clad sheet, and subjected to endurance tests, thermal-shock tests, and erosion tests in a turbojet engine. The results of the endurance tests are considered highly encouraging, and the bond between the cladding and the molybdenum appeared to be uniformly good after thermal cycling, though the tear strength was appreciably lower. In the erosion tests on molybdenum and high-temperature-alloy nozzle vanes both materials were severely attacked.

Of the diffusion barriers tested, the nickel and 'Monel' both improved the bond strength of the as-rolled sheet and appeared to have a beneficial effect on workability. Palladium, copper and chromium barriers were the only ones to prevent the occurrence of hard intermetallic compounds.

The paper includes a short section on experience gained in fabricating various turbine components from clad sheet.

Forging of Molybdenum and Its Alloys

J. J. RUSS: 'Forging of Molybdenum and Its Alloys.' Contained in *Fabrication of Molybdenum*, pp. 169–73. Published by *Amer. Soc. Metals*, 1959.

The paper is divided into two sections concerned, respectively, with the forging problems resulting from volatilisation of molybdenum at temperatures above 1200°F. (650°C.), and the need for the development of testing techniques which would ensure adequate control during processing.

In the author's experience volatilisation entails

the ancillary problem of the saturation of the furnace (especially controlled-atmosphere furnaces) with molybdenum trioxide, which can have deleterious effects on materials which are subsequently processed. Alloys containing more than 3 per cent. molybdenum are liable to catastrophic oxidation; the 18-8 and straight-chromium stainless steels, low-alloy chromium - molybdenum steel, and 'Hastelloy C' are subjected to rapid oxidation; and a slight increase in oxidation occurs in materials having high nickel and chromium contents. The following specific cases are referred to: '16-25-6' forgings exhibited severe spalling and catastrophic oxidation; 'S-816' exhibited severe pitting and oxidation; 'Inco 700' exhibited an oxidation rate higher than normal and some pitting; 'Inconel X', which contains no molybdenum, was unaffected.

Brazing of Molybdenum with Nickel-containing Alloys

G. S. HOPPIN: 'Brazing of Molybdenum.'

Contained in *Fabrication of Molybdenum*, pp. 77-87. Published by *Amer. Soc. Metals*, 1959.

The successful use of molybdenum for high-temperature applications will, in many cases, be contingent on the degree to which the component can be fabricated by brazing. This paper 'reviews the available brazing processes for molybdenum, the key problems, and past and present work on brazing the metal for low- and high-temperature service'.

References in the literature show that molybdenum can be brazed by any of the usual heating processes (e.g., by induction, torch, furnace and resistance techniques) but that furnace brazing, the method used in most of the work described by the author, must be carried out *in vacuo* or in an inert or reducing atmosphere. The main problems are metallurgical: (1) the tendency of unalloyed molybdenum to recrystallise at temperatures above 1800°F. (980°C.); (2) the lowering of the recrystallisation temperature of the alloyed grades (which is much higher than that of the pure metal) as a result of diffusion into the alloy of atoms of the brazing material; (3) the formation of intermetallic compounds between the molybdenum and the brazing material; (4) the relative weakness of brazing materials at the elevated temperatures at which the strength of molybdenum offers most advantages. Brazing of components for 'low-temperature' service (i.e., below 1200°F. : 650°C.) offers no difficulties since none of these metallurgical problems is involved. Consequently the major portion of the paper is concerned with work on the development of brazing materials which would be suitable for use with a molybdenum-titanium alloy containing ½ per cent. titanium and which would satisfy the requirements of 'high-temperature' service (i.e., in a range which was arbitrarily fixed at 1200°-3000°F. (650°-1650°C.)).

Simple lap-joints were furnace-brazed (under a hydrogen atmosphere) from molybdenum-titanium-alloy sheet, using the following brazing materials: 86 Fe-14 Si; 75 Fe-14 W-12 Si; copper; 95 Ag-5 Cu; 94 Ni-6 Si; 48 Ni-48 Cr-4 Si; 60 Pd-40 Ni; 65 Pd-35 Cu; 70 Pd-30 Cu; 93 Pd-7 Al; 70 Ni-20 Cr-10 Si; 86 Ni-7 Cr-4 Si-3 B. The specimens were then subjected to shear tests at room temperature and/or 700°, 1000° or 1500°F. (370°, 548° or 815°C.).

The room-temperature tests showed the strongest filler materials to be copper and the palladium-copper and silver-copper alloys. Only the palladium-copper alloy, however, possessed a melting point suitable for elevated - temperature service. High - temperature tests on the palladium-base alloys showed the palladium-40 nickel alloy to be the strongest alloy and the palladium-35 copper the weakest. The results obtained with the palladium-base brazing materials are considered to be encouraging. Brief reference is, however, made also to another promising approach employed in work on molybdenum turbine blading: the use of nickel-base high-temperature brazing alloys applied over a chromium diffusion barrier.

Ultrasonic Welding of Molybdenum and Nickel-containing Materials

J. B. JONES: 'Ultrasonic Welding.'

Contained in *Fabrication of Molybdenum*, pp. 88-102. Published by *Amer. Soc. Metals*, 1959.

Ultrasonic welding, a method of bonding similar or dissimilar metals by the introduction of vibratory energy into the metals in the area to be bonded, is reviewed in relation to (1) the apparatus and procedures used, (2) its capabilities and the advantages it offers, (3) the metallurgical and thermal phenomena involved, (4) joining of dissimilar metals, and (5) welding of molybdenum.

It is stated that ultrasonic welding can be employed to fabricate, in gauges up to about 0·03 in. (0·75 mm.), such materials as niobium, tantalum, zirconium, titanium, 'Inconel', 'Inconel X' and '17-7 PH' stainless steel, and the section on welding of dissimilar materials includes reference to the production of joints between Type 316 18-8-Mo stainless steel and 'Zircaloy 2', 'AM-350' stainless steel and aluminium, nickel and low-carbon steel, nickel and gold-plated 'Kovar', tungsten and Type 302 stainless steel, and tungsten and nickel-plated low-carbon steel. Comparative data are presented on the strength of welds joining molybdenum to molybdenum, and molybdenum to molybdenum using nickel or platinum foil as filler metals.

The following features characteristic of ultrasonic welds are discussed: interfacial disturbance, which is not accompanied by significant reduction in load-carrying capacity resulting from over-all deformation; transformations indicative of substantial rises in temperature and accompanied by unusual metallurgical structures; and recrystallisation.

Steel for Reactor and Processing Plants

B. WATKINS: 'Steel for Reactors and Processing Plants.'

Nuclear Engineering, 1959, vol. 4, July/Aug./Sept., pp. 296–303.

The development of gas-cooled reactors has necessitated the construction of pressure vessels, up to 70 ft. (21 m.) in diameter, in 2–4 in. (5–10 cm.) steel plate, the site-welding of which has involved difficulties. At present the choice of steel for this purpose is restricted to aluminium- or silicon-killed mild steel, but the trend towards higher gas temperatures will require either the use of alloy steels for, or some form of insulation or cooling of, the reactor shell. A third group of steels, the chromium-nickel austenitic stainless steels, find application not only in reactor construction but also in plant processing uranium and plutonium. In this paper (in which reference is made to a *bibliography* of 48 items) the author reviews the properties of the mild, low-alloy and stainless steels at present in use in, or under development for, reactors and processing plants.

The discussion and data presented relate to mild steels of various composition, low-alloy chromium-molybdenum steels, and stabilised and unstabilised chromium-nickel stainless steels of 18-8 and 18-12 type. The steels are considered in sections dealing with mechanical (tensile and creep) properties, chemical compatability (with carbon dioxide, aqueous media, liquid metal, and nitric acid), welding characteristics, and the effects of irradiation.

The paper includes two data sheets: the first summarises the physical, chemical and mechanical properties of the steels discussed and the effects, on these properties, of irradiation; the other contains a series of creep design curves.

Corrosion of Austenitic Stainless Steel by Fuel-Oil Ash

H. L. LOGAN: 'Corrosion of Type 310 Stainless Steel by Synthetic Fuel-Oil Ash.'

Corrosion, 1959, vol. 15, Aug., pp. 443t–6t.

The investigation described was carried out to determine the minimum temperature at which A.I.S.I. Type 310 austenitic stainless steel is susceptible to attack by fuel-oil ash, and formed the first phase of a study of fuel-oil-ash corrosion of the steel which is in progress at the U.S. National Bureau of Standards. Investigation of the corrosion mechanism is continuing.

The composition of the steel is given as: carbon 0·06, silicon 0·19, manganese 1·53, phosphorus 0·022, sulphur 0·005, chromium 24·8, nickel 21·2, per cent. In the initial stages of the investigation tests were carried out with a synthetic oil-ash mixture of 85 per cent. V_2O_5+15 per cent. Na_2SO_4, but most of the data reported were derived from tests with V_2O_5+$NaVO_3$ mixtures. The synthetic oil-ash mixture selected for study was placed in a tube fabricated from Type 310 stainless steel. The tube was then sealed and subjected in a furnace to a temperature gradient within the range in which incipient attack was expected to occur. Six thermocouples welded at intervals to the outside surface of the tube (data derived from which were supplemented by the results of metallographic examination and measurement of the wall thickness of the tube after testing) permitted determination of the minimum temperature at which the ash mixture attacked the steel. In most of the experiments moist air was allowed to flow through the tube, but, in one, moist helium was used instead of moist air. A few tests were run in which V_2O_5 or $NaVO_3$ were exposed separately to the steel in an atmosphere of wet or dry air or dry helium.

The results, which are supplemented by photomicrographs of the specimens after exposure, are discussed in sections relating to the minimum temperatures at which the steel was attacked; microstructures at the steel/slag interface; changes in the gas content of sealed tubes; identification of compounds present in the slag formed in the tubes during testing. The conclusions drawn from these results are quoted below.

'(1) A mixture of 67 per cent. V_2O_5+33 per cent. $NaVO_3$ attacked the steel at 1075°F. (580°C.). Mixtures of V_2O_5+$NaVO_3$ containing 34 per cent. and 88·5 per cent. of V_2O_5 attacked the steel at 1165°F. (630°C.) and 1220°F. (660°C.), respectively. A mixture of 85 per cent. V_2O_5+15 per cent. Na_2SO_4 attacked the steel at 1115°F. (600°C.).

'(2) An unidentified phase structure developed at the steel/slag interface, and penetrated into some grain boundaries at this interface, on heating V_2O_5, $NaVO_3$, V_2O_5+$NaVO_3$, or V_2O_5−Na_2SO_4 in a steel tube at temperatures in the range 1100°F. to 1850°F. (595° to 1010°C.). The maximum temperature at which this phase occurred at the interface varied with the vanadium content of the compound or mixture, ranging from 1450°F. (785°C.) for $NaVO_3$ alone to 1850°F. (1010°C.) for V_2O_5.

'(3) At high temperatures the unidentified phase penetrated into the steel along the grain boundaries, and, in some instances, completely surrounded grains of steel.

'(4) An unidentified phase was found in the steel heated above 1700°F. (925°C.) in contact with a vanadium compound.

'(5) The slag contained some components of the steel in approximately the same proportions as they were found in the steel. X-ray diffraction patterns of the slag indicated that it contained $NiO.V_2O_5$, $2NiO.V_2O_5$, $CrVO_4$, $Fe_2O_3.2V_2O_5$, and possibly $FeVO_4$.

'(6) The slag obtained by heating a mixture of 33 per cent. $NaVO_3$+67 per cent. V_2O_5 conducted electricity in both the liquid and solid state.'

Problems Involved in the Operation of Peat-Fired Gas Turbines

'Studies in the Chemistry of the Operation of Solid-fuel-fired Gas Turbines.'

J. A. WADDAMS and J. C. WRIGHT: '1. Pulverised Peat as an Open-cycle Gas-Turbine Fuel.'
Jnl. Inst. Fuel, 1959, vol. 32, Apr., pp. 178–81.

J. A. WADDAMS and J. C. WHITE: '2. An Investigation of Pulverised Peat and its Combustion in a Rig Simulating Conditions in an Open-cycle Gas Turbine.'
Ibid., pp. 182–90.

J. A. WADDAMS and J. C. WHITE: '3. Effect of Operating Variables on the Rate of Ash Deposition during Short Runs with a Pulverised-Peat Combustion Rig.'
Ibid., pp. 190–4.

J. A. WADDAMS and J. C. WHITE: '4. Examination of Ash Samples from an Open-cycle Gas Turbine after Running on Pulverised Peat.'
Ibid., 1959, vol. 32, May, pp. 241–5.

J. A. WADDAMS, J. C. WHITE and P. S. GRAY: '5. Dry Corrosion Aspects of Peat-Ash Deposition.
Ibid., pp. 246–9.

J. A. WADDAMS and J. C. WHITE: '6. A Proposed Mechanism for the Deposition and Bonding of Ashes in an Open-cycle Peat-fired Gas Turbine.'
Ibid., pp. 249–52.

The six papers present the results of a comprehensive investigation of the causes and mechanism of corrosion, erosion, and ash deposition in a peat-fired open-cycle gas turbine. Laboratory experiments were supplemented by work with a large combustion rig designed to simulate conditions in a peat-fired turbine, and, in addition, research was carried out, using a full-scale development gas turbine, on peat fuel, ash, and turbine blades. The combustion chamber of the testing rig and turbine comprised a short refractory-lined section at the burner end and a flame tube of 'Nimonic 75'. Turbine blades were of 'Nimonic 80A'.

The first paper describes the characteristics of peat as a fuel, and outlines the difficulties anticipated in the turbine from one or more of the phenomena of ash deposition, erosion, and corrosion. Previous work in the same field is also reviewed. The characteristics of the two peats used in the investigation and their ashes are examined in detail in part 2. It became evident that ashes resulting from combustion of peat under laboratory conditions could differ appreciably from those produced by combustion in a full-scale turbine, and to enable investigation of the latter type of ash without resorting to full-scale engine runs, use was made of a combustion rig, which is also described in the second paper of the series. The main operating variables of the rig are discussed in the third paper. Using similar methods to those employed in examination of laboratory-produced and rig-produced peat ash, a number of ashes and deposits from the full-scale turbine were examined after operation under a variety of conditions: the results are presented in paper 4. Evaluation of the data gained as a result of these tests, together with the results of an investigation of dry corrosion of the turbine blades (see paper 5), leads the authors to propose a mechanism for the deposition and bonding of ash in the open-cycle peat-fired gas turbine. The mechanism advanced is outlined in paper 6 of the series.

The information given in paper 5, that dealing with dry corrosion of 'Nimonic 80A' turbine blades, is closely akin to that given in a paper by WADDAMS and WRIGHT in *Metallurgia*, 1959, vol. 59, Apr., pp. 161–4: see abstract in *Nickel Bulletin*, 1959, vol. 32, No. 7, p. 227.

Notch-Sensitivity Effects in Stress-Corrosion Testing of High-Strength Steels

B. F. BROWN: 'Notch-Sensitivity Effects in Stress Corrosion and Hydrogen-Embrittlement Tests on High-Strength Steels.'
Corrosion, 1959, vol. 15, Aug., pp. 399t–402t.

There are no universally accepted tests for determining susceptibility of a steel to stress-corrosion cracking or to hydrogen embrittlement. In most cases an evaluation of susceptibility is sought in tests in which, if complete fracture of the specimen occurs, the measurement reported is the time-to-rupture. The purpose of the present paper is to show how, in some instances, this test procedure can give an erroneous impression of the rate of cracking by stress corrosion or by hydrogen embrittlement.

The author first considers the fracture behaviour of high-strength steels under purely mechanical conditions in the presence of a stress-raiser, and emphasises the important rôle played by the degree to which the composition, heat-treatment, size and geometry of the specimen renders the steel notch-sensitive. He concludes that failure of a notch-resistant specimen in a stress-corrosion test under constant load might be expected to require a far greater degree of stress corrosion than that involved in the failure of a notch-sensitive specimen under the same conditions. To assess the validity of these assumptions the author then refers to fracture data derived from stress-corrosion tests on straight-chromium stainless-steel specimens tempered at 875° or 1075°F. (470° or 580°C.) and shows that, under certain conditions, a great part of the fracture occurring during the test is attributable to purely mechanical effects.

The general conclusions drawn from these results are that, for the materials used, rupture of a given steel after a short period of exposure would appear a sufficient criterion, without distinguishing between stress-corrosion cracking and brittle fracture, for the practical purpose of rejecting the material. Attention is, however, directed to the fact that this procedure could sometimes lead to rejection of an extremely notch-sensitive material which cracks by stress-corrosion only very slowly, and which in other

geometries and stress configurations might not be susceptible to brittle fracture. That no cracking occurs in specimens of a given material would suffice for tentative acceptance of the material from the standpoint of stress-corrosion cracking, although such a result throws no light upon notch-sensitivity.

Factors Influencing the Susceptibility of Copper Alloys to Stress-Corrosion Cracking

D. H. THOMPSON: 'Probability as Related to Stress-Corrosion Cracking of Copper Alloys.'
Corrosion, 1959, vol. 15, Aug., pp. 433t-6t.

In the introduction to the paper the author discusses the six factors determining the probability of stress-corrosion cracking in copper alloys: the concentration of ammonia, water and air present, the level of the residual or applied stress to which the alloy is subjected, the time for which the alloy is exposed to these conditions, and the degree to which a given alloy is susceptible to stress corrosion.

The probability of occurrence of stress corrosion is expressed mathematically in terms of these factors, and the contribution of each is then discussed in relation to specific case histories of stress-corrosion cracking. The failures discussed occurred in red-brass pipe, cold-drawn copper water tubing, 70-30 cupro-nickel tubing, Admiralty- and aluminium-brass condenser tubes, and yellow-brass screw shells.

Finally reference is made to the two factors which may be most readily modified as a means of reducing susceptibility to cracking: residual stress and the alloy itself. The efficacy of stress-relief annealing is briefly considered, and, in discussing the selection of a suitably resistant alloy, the author classifies those available in the following ascending order of resistance: brass containing over 20 per cent. of zinc, and brass containing over 20 per cent. of zinc plus small amounts of lead, tin or aluminium; brasses containing less than 20 per cent. of zinc, aluminium bronze, nickel silver, phosphor bronze; silicon bronze, phosphorised copper; cupro-nickel, tough-pitch copper.

Stress-Corrosion Cracking of Oil-Production Tubing

R. L. MCGLASSON and W. D. GREATHOUSE: 'Stress-Corrosion Cracking of Oil-Country Tubular Goods.'
Corrosion, 1959, vol. 15, Aug., pp. 437t-42t.

Stress-corrosion cracking of oil-production tubing is reviewed in relation to (1) sulphide-corrosion cracking, and (2) cracking in the presence of sweet environments. Sulphide corrosion was studied in terms of the environmental factors and the minimum hardness and applied-stress limits which induce cracking. Work on stress corrosion in sweet environments was directed towards determining the significant constituents of the environments contributing to cracking, and, in particular, towards establishing whether sulphide is present in such environments and, if so, the critical concentrations necessary for occurrence of cracking.

The authors' experiments were carried out mainly on two tube steels ('N-80' and 'J-55') but the results obtained from these tests are supplemented by other data, culled from the literature, on, *inter alia*, various grades of nickel and chromium steels. Details are given of a 'notched-ring' stress-corrosion test which is claimed to give more reproducible results than those obtained with polished-beam specimens and which provided a means of determining hardness/applied-stress limits at stresses below the yield stress. The paper also includes an evaluation, based on reports in the literature, of the susceptibility of various grades of steel to cracking in oil-well environments.

The authors' assessment of the data reviewed leads them to draw the following conclusions:

'(1) All types of steel will crack in aqueous systems containing H_2S.

'(2) There are hardness and applied-stress minima below which a given steel will not crack, and these minima are inversely related.

'(3) For given strength levels, low-chemistry quenched and tempered steels are less susceptible to cracking than alloy steels.

'(4) Sweet corrosion-cracking failures have not been produced in laboratory environments free of sulphide.

'(5) Some sweet condensate fields where cracking has occurred in tubing contain sufficient quantities of sulphide to enter into the corrosion process.

'(6) Cracking is produced in solutions containing 15·2 p.p.m. H_2S and less.

'(7) Time to cracking failures is inversely related to the concentration of H_2S in solution.'

Friction Behaviour of Austenitic Materials During Deep Drawing

H. WIEGAND and K. H. KLOOS: 'Influence of the Surface Boundary Layer on the Friction Behaviour of Austenitic Materials during Cold Deformation Involved in Deep Drawing.'
Metalloberfläche, 1959, vol. 13, Aug., pp. 229-33.

The paper gives details of an experimental technique which permits investigation of the frictional phenomena involved during deep drawing of austenitic materials, and of their relationship to boundary lubrication and wear processes.

During deep drawing the surface of the material is subjected to plastic deformation (the degree of which is governed by the lubricating agent employed) and hence to a microstructural ($\gamma \rightarrow \alpha$) change. The amount of martensitic constituents present in the boundary layer may be determined by means of X-ray techniques, and the γ-α distribution so determined then offers a criterion by which to assess the lubricating characteristics of the high-pressure lubricating substance used.

The data reported in the paper illustrate the application of the technique to an 18-8-type chromium-nickel stainless steel. Study of the transformation behaviour of this steel during deep drawing demonstrated the superior lubricating qualities of chemical transformation (oxalate) coatings compared with stable organic soap compounds.

Cleaning and Passivation of Stainless Steels

L. LOWRY and J. THOMPSON: 'Cleaning and Passivation of Corrosion-Resisting Steels.'

U.S. Office of Technical Services, Naval Gun Factory, Tech. Rept. NGF-T-28-57, 1959; 21 pp.+ tables. *PB* 131964.

The object of the investigation was two-fold: to study requirements with respect to cleaning and passivation of stainless steels, and, on the basis of the findings, to prepare, if necessary, a specification covering passivation requirements.

The scope of the investigation included:

(1) A survey (which is presented in the report) of methods employed to remove surface contaminants such as oil or grease; scale; welding, soldering or brazing flux or slag; metallic lubricants such as copper, lead or cadmium applied to stainless-steel wire for cold heading, wire drawing, or spring winding; or iron embedded in the surface as a result of finishing operations such as tumbling, polishing, grinding, sand and shot blasting, machining, forming or drawing.

(2) A study of the dimensional changes occurring in the following grades of stainless steel (in the annealed condition or heat-treated to various hardnesses or to produce sensitisation) as a result of exposure to nitric-acid passivating solutions or to nitric-acid/hydrofluoric-acid pickling solutions: straight-chromium martensitic and ferritic steels (A.I.S.I. Types 410, 420, 440, 405 and 430); stabilised and unstabilised chromium-nickel stainless steels (A.I.S.I. Types 321 and 302); manganese-nickel-chromium steels (A.I.S.I. Types 201 and 202). The effects of chloride contamination of nitric-acid passivating solutions were also determined.

(3) An evaluation of methods suggested for determination of iron contamination on the surface of finished stainless-steel articles.

In general the corrosion suffered by all three groups of steel when exposed to nitric-acid passivating solutions was low, varying from $0{\cdot}17\times10^{-6}$ to $4{\cdot}51\times10^{-6}$ in./unit area/hour. Contamination of the solution by approximately 0·4 wt. per cent. of chlorides slightly increased the rate of attack on steels of the A.I.S.I. Type 200 and 300 series; corrosion of the A.I.S.I. Type 400 steels was considerably increased, the corrosion rates ranging from 775×10^{-6} to 3446×10^{-6} in./unit area/hour. Use of commercial nitric acid containing traces of chlorides caused no significant increase in corrosive attack.

Corrosion resulting from the steels' exposure to nitric-acid/hydrofluoric-acid pickling solutions was much greater than that produced by the nitric-acid passivating solutions. Corrosion rates ranged from $32{\cdot}8\times10^{-6}$ to $310{\cdot}2\times10^{-6}$ in./unit area/hour for steels of the A.I.S.I. Type 200 and 300 series, and from 1904×10^{-6} to 6702×10^{-6} in./unit area/hour for the straight-chromium steels of the A.I.S.I. Type 400 series.

Sensitisation increased the corrosion rate of Type 302 steel in the nitric-acid passivating solution from $0{\cdot}51\times10^{-6}$ to $55{\cdot}1\times10^{-6}$ in./unit area/hour. In nitric-acid hydrofluoric-acid pickling solutions the rate of attack rose from $74{\cdot}4\times10^{-6}$ to 3399×10^{-6} in./unit area/hour. The titanium-stabilised Type 321 steel, exposed to the same heat-treatment as the Type 302 steel without evidence of carbide precipitation, exhibited a very low rate of attack in the passivating solutions, and in the pickling solution attack was relatively slight.

The following two methods, applicable to all types of stainless steel, are considered to be the most satisfactory for determination of iron contamination: exposure of the steel to 100 per cent. humidity at 100°F. (38°C.); alternate wetting in tap water and drying, to produce rust where free iron is present. Chemical methods of detection such as the potassium-ferricyanide/nitric-acid test and the copper-sulphate test are applicable only to the austenitic chromium-nickel steels and to the manganese-nickel-chromium steels.

Slip Casting of Stainless Steels

H. HAUSNER: 'Slip Casting: A New Method in Powder Metallurgy.'

Paper to *Third Plansee Seminar*, July 1958.
Plansee Proceedings, 1958, pp. 419-33; disc., pp. 434-7.
Published by *Metallwerk Plansee A.G.*, 1959.

Conventional powder-metallurgy techniques possess disadvantages which considerably restrict the range of their application. Their scope is, for example, limited by the capacity of conventional presses, by their inability (due to non-uniform pressure distributions in the mass of metal powders) to produce components of complicated shape, and by the fact that they are economic only on the basis of relatively large-scale production. In the field of ceramics, however, these disadvantages have to a great extent been overcome by the development of the slip-casting process, which eliminates the need for application of pressure. (In ceramics a slip or slurry is defined as a fluid suspension of ceramic powders in liquids, and slip casting refers to the filling of a plaster-of-paris mould, a negative of the desired shape, with this slip. As a result of the capillary effect of the pores, the liquid penetrates into the plaster mould and the remaining ceramic powder particles adhere strongly in the mould during the drying operation. The slip for casting is prepared from water, ceramic powders such as clay and talc,

and a small amount of deflocculant, the latter being added in order to prevent, or rather to delay, the settling of the powder particles and to create the desirable viscosity of the slip. After partial drying in the mould and final drying in air at room- or elevated temperature, the moulded ceramic mass is then fired.) In this paper the author describes the application of slip casting to metal powders.

In a general description of the technique the processing steps are listed, and variables are considered (the most important being the viscosity and pH of the slip) which have hitherto been outside the province of the powder metallurgist. Casting moulds are discussed, a comparison is made of the properties of individual powder particles in a powder mass produced by conventional powder metallurgy and by slip casting, and the mechanisms governing the movement of powder particles in the slip are briefly noted.

The main section of the paper is devoted to a detailed description of the procedure and variables involved in slip casting 18-8-Mo (Type 316) stainless steel. Details are given of: the particle size distribution and the apparent density of the powders; the properties of the deflocculants used; experiments to determine the relationships between the viscosity and the pH of the slip, and between the liquid/solid ratio of the slip and the physical properties of the slip-cast stainless steel in the as-dried and as-sintered conditions; and shrinkage during sintering.

The paper also contains a short description of the application of the slip-casting process to production of stainless-steel/UO_2 cermets and of molybdenum and tungsten components, and examples are given of articles produced by the process (including stainless-steel turbine blades).

The author emphasises that slip casting is not intended to replace conventional pressure-compacting techniques, but rather to extend the applications of powder metallurgy to production of large and complicated shapes, and to enable economic small-scale production of components for which conventional techniques are not suitable.

Direct Electron-Microscopical Examination of Thin Metallic Foils

C. BEAUVAIS: 'Application of the Electron Microscope to Direct Examination of Thin Metal and Alloy Foils.'

Métaux, 1959, vol. 34, June, pp. 247-57; July-Aug., pp. 291-301.

The application of the electron microscope to direct examination of metal or alloy foils (i.e., without use of replicas) is hindered by the absence of a simple established technique for production of the very thin specimens required. In this paper the author describes the methods which he has employed in the preparation of foil specimens of various materials, and presents the micrographic results obtained from direct electron-microscopical examination.

The paper is divided into two parts. The first is sub-divided into sections dealing, respectively, with the general principals involved in the examination of metallic foils by means of the electron microscope, and with the three methods used by the author to prepare foils sufficiently thin for this purpose: mechanical polishing, mechanical deformation, and electrolytic polishing. Full details of the three techniques are given. In outlining these, reference is made to electrolytes suitable for use in polishing *inter alia*, nickel, 18-8 steel, and 'Nimonic' alloys.

The second part of the paper exemplifies, by reference to numerous photomicrographs, the results obtained by direct electron-microscopical study of foils produced by the techniques previously discussed. The photomicrographs illustrate the application of the technique to study of: dislocations and carbide precipitation in 18-8 chromium-nickel stainless steel; the structures of carbon steel, and of a 90-10 copper-aluminium alloy and a copper-beryllium alloy in various conditions of heat-treatment; precipitation reactions in a nickel-chromium-cobalt-titanium-aluminium alloy during ageing; precipitation reactions in 'Duralumin'.

Techniques for Growing Single Crystals of Austenitic Stainless Steel

R. D. LEGGETT, R. E. REED and H. W. PAXTON: 'Growth of Single Crystals of Stainless Steel.'

Trans. Metallurgical Soc. Amer. Inst. Mechanical Engineers, 1959, vol. 215, Aug., pp. 679-80.

In this note the authors give details of techniques which have been developed for growing monocrystals and bicrystals of 20 per cent. chromium and 20-20 and 18-8 chromium-nickel stainless steels. Two methods are described: crystal growth by strain-annealing and crystal growth from the melt.

Determination of Magnetic Phases Present in Steels

P. BASTIEN and A. SULMONT: 'The Value of Testing in a Strong Magnetic Field and its Application to Study of Steels.'

Mémoires Scientifiques de la Revue de Métallurgie, 1959, vol. 56, July, pp. 131-42; disc., p. 143.

The apparatus described in the paper can be used to measure the intensity of magnetisation exhibited by cylindrical specimens (100 mm. long and 6 mm. in diameter) in a magnetic field of from 100-3,500 Oe. at temperatures in the range 20°-700°C. It therefore permits quantitative determination of magnetic phases present in steels at temperatures within this range, and hence provides a non-destructive method of determining the amount of residual austenite present in steels during heat-treatment or at room temperature.

The paper is divided into three sections. In the first the authors outline formulae relating variations in magnetisation to the magnetic field, the temperature, and the amount of ferromagnetic phase present. From the values obtained in fields of from 1500-4000 Oe the magnetisation of the steel

can be calculated, which then, knowing the magnetic saturation of the specimen were it 100 per cent. ferromagnetic, enables deduction of the amount of non-magnetic phase (i.e., residual austenite) present.

The second section contains a detailed description of the apparatus used. In the third the authors give particulars of experiments which were carried out to verify the theoretical formulae developed in the first section, and to evaluate the scope of, and the procedures to be used with, the apparatus. Tests were conducted on Armco iron and on chromium-nickel steels of 18-2, 18-4 and 18-6 type. Data are presented illustrating the application of the apparatus to determination of the percentage of ferrite present in an 18-4 chromium-nickel steel as a function of holding time at various temperatures.

Welding of Ferritic to Austenitic Tubing

B. LÖFBLAD and G. LINDH: 'Development of a Transition Weld Between Ferritic and Austenitic Superheater Tubing.'

Welding and Metal Fabrication, 1959, vol. 27, Aug./Sept., pp. 325-30, 349.

The paper is an English translation of that presented by the authors at the 1958 Annual Assembly of the International Institute of Welding, Vienna; see abstract in *Nickel Bulletin*, 1959, vol. 32, No. 3, p.96.

Joining of Dissimilar Materials

J. G. YOUNG and A. A. SMITH: 'Joining Dissimilar Metals.'

Welding and Metal Fabrication, 1959, vol. 27, July, pp. 275-81; Aug./Sept., pp. 331-9.

The review is in two sections. In the first section the authors discuss the processes available for joining dissimilar metals and draw attention to the problems involved in, and the metallurgical factors which govern, the production of a satisfactory joint. In the second section the practical significance of these general principles is illustrated by reference to particular problems which confront the metallurgist in joining various dissimilar materials, and to the methods by which they may be overcome.

Part I of the paper opens with a short review of the factors influencing the selection of the joining method (the type of joint, its shape and thickness, the conditions to which it will be subjected in service). The joining processes available (argon-arc welding, metal-arc and inert-gas metal-arc welding, gas-welding, brazing and braze-welding) are discussed in relation to the applications for which they are mainly suitable, and attention is then drawn to the general metallurgical principles involved in joining dissimilar materials. In the latter connection, factors influencing the constitution and microstructure of such joints are outlined in sub-sections dealing with: alloying, the interpretation and use of equilibrium diagrams, and the properties of the various phases which may be present in a joint; hot-cracking; dilution during fusion welding or brazing, and the uniformity of composition of the weld or braze. Finally the authors describe, in some detail, the principles of indirect welding techniques, brazing, solid-state bonding and resistance welding, and consider the particular advantages they offer in joining dissimilar metals.

Part II of the paper is concerned with a discussion of the problems involved in joining materials representative of the systems referred to below. In each case the problems and their solutions are particularised in relation to specific case histories, the authors' notes on which are supplemented by photomacrographs or photomicrographs of the joints.

Joining of Copper to Nickel

Reference is made to an argon-arc butt-weld between phosphorus-deoxidised copper and nickel.

Joining of Nickel and Nickel Alloys to Steel

The general discussion is exemplified by notes on the welding of nickel to mild steel, 'Nimonic 75' to 18-8 stainless steel, and 'Inconel' to mild steel.

Joining of Dissimilar Alloy Steels

The Schaeffler diagram and its use in predicting, from the composition of the basis and filler metals, the approximate proportions of austenite and ferrite in the weld metal are discussed, and its importance in selecting a filler metal which will prevent cracking is illustrated in connection with the production of lap joints between 18-8 and mild steels, and welding of mild steel clad with stainless steel.

Joining of Aluminium to Copper and to Steel

Joining of Copper and Copper Alloys to Steel

Methods of overcoming dilution and other problems encountered in joining two groups of materials are exemplified by reference to the procedures employed in welding, *inter alia*, aluminium-bronze and stainless steel, and in brazing mild steel to 'Nimonic 75', and 'Kovar' to mild steel.

Techniques suitable for joining dissimilar combinations of the following materials are summarised in two tables concerned, respectively, with fusion welding and brazing techniques: low-alloy steels (film-forming and non-film-forming), high-alloy and stainless steels and irons, nickel, copper-bearing nickel alloys, nickel alloys, copper, copper alloys, aluminium-bronze, titanium, and aluminium alloys.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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